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ERDEC-TR-246

# INDEPENDENT VERIFICATION OF AN ERDEC RESEARCH PROGRAM RECOMMENDING EMERY 3004 (ETHYLFLO 164) AS A SAFE DOP REPLACEMENT FOR MASK AND FILTER TESTING



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**13. ABSTRACT** *(Maximum 200 words)*
In April, 1986, the U.S. Army's Office of the Surgeon General (OTSG) took the position that dioctyl phthalate (DOP) was a suspected carcinogen. The U.S. Army had used DOP for many decades in non-destructive serviceability testing of respirator canisters and protective filters, and in a variety of aerosol penetration studies including mask leakage and face fit. A program was initiated in September 1987 to find a safe replacement material for DOP, under sponsorship of the Product Assurance Directorate (PAD). A synthetic lubricant named "Emery 3004," from the class of compounds called poly-alpha olefins (PAOs), was approved by the OTSG on 8 January 1992 for use Army-wide as a safe replacement for DOP in "hot smoke" and "cold smoke" testing machines. Subsequently, ERDEC scientists recommended Emery 3004 as a general replacement for DOP, and interest among other users grew rapidly until, today, Emery 3004 is known to virtually all filter testers. Among these are the Department of Energy, and the pharmaceutical organization of Eli Lilly and Company. This report presents the independent results of DOE and Lilly in their own studies of Emery 3004, their verification of ERDEC data, and their intention to replace DOP with Emery 3004 in their own operations.

**14. SUBJECT TERMS**

| | | | | | |
|---|---|---|---|---|---|
| DOP | Cost | Toxicity | Respirators | "Q-testers" | "Emery 3004" |
| PAOs | Masks | TDA-100 | Hot smokes | Carcinogens | DOP replacement |
| Q-127 | Face-fit | Mutagens | Test smokes | Cold smokes | (Continued on page 2) |

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## PREFACE

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## CONTENTS

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# INDEPENDENT VERIFICATION OF AN ERDEC RESEARCH PROGRAM RECOMMENDING EMERY 3004 (ETHYLFLO 164) AS A SAFE DOP REPLACEMENT FOR MASK AND FILTER TESTING

## 1. INTRODUCTION

### 1.1 Background

The U.S. Army routinely performs 100% quality control testing of filter canisters manufactured for use with field-issue gas masks, and periodic sampling and testing of canisters stored in its supply depots. In April, 1986, the U.S. Army's Office of the Surgeon General (OTSG) announced that DOP posed potentially serious health risks to workers, and placed severe restrictions upon testing with it; agencies were also informed that dioctyl sebacate (DOS) would no longer be acceptable as a DOP replacement material, and that similar restrictions would apply for both. These restrictions included occupational exposure monitoring of workers exposed to DOP aerosols and liquid, medical surveillance, issue of personal protective equipment, formal notification to workers of associated risks, and labeling of work areas as "cancer suspect agent areas."

The above actions placed severe restrictions upon routine, 100% quality assurance testing of filters and other equipment. For this reason, in 1987 the U.S. Army initiated a detailed study of the problem of finding an acceptable substitute material for DOP that could meet all standard military test specifications while itself being a non-carcinogen and, ideally, having other attributes including acceptable acute inhalation toxicity, low cost, ready availability, and the ability to replace DOP directly in machines at test installations without retrofit or other modification of these machines.

The Army had used DOP for many decades in non-destructive servicability testing of respirator canisters and protective filters, and in a variety of aerosol penetration studies including mask leakage and face fit. The program initiated in September 1987 to find a safe replacement material for DOP was sponsored by the Product Assurance Directorate (PAD).[1,2] A synthetic lubricant named "Emery 3004," from the class of compounds called poly-alpha olefins (PAOs), was approved by the OTSG on 8 January 1992 for use Army-wide as a safe replacement for DOP in "hot smoke" and "cold smoke" testing machines. Emery 3004 was approved after successfully passing three tiers of mutagenicity testing that included the Ames system assay, the sex-linked recessive lethal test in fruit flies, and the rodent bone marrow micronucleus assay performed with rats.

A less-viscous PAO, "Emery 3002," also was also approved by the OTSG on 24 February 1993 for Army-wide use in cold-smoke applications (Reference 3). The Ethyl Corporation[4] is the primary manufacturer of these PAOs, under the trade names "Ethylflo 162" (repackaged as Emery 3002), and "Ethylflo 164" (repackaged as Emery 3004). These materials are extremely useful DOP replacements. They perform at least as well as DOP in various testing machines. They can replace DOP directly in existing machines without modification. They are inexpensive, readily available, and should continue to remain so in the future. Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are readily specifiable, non-corrosive, free of natural impurities, thermally and chemically stable, and safe to work with. They are recommended to replace DOP in Army-wide testing as soon as is practicable.

## 1.2 U.S. Department of Energy (DOE)

The Westinghouse Hanford Company, under Contract DE-AC06-87RL10930, is the operations and engineering contractor for the old Hanford Nuclear Reservation near Richland, Washington. These operations include the careful inspection of high-efficiency particulate aerosol (HEPA) filters which are depended upon to keep particulates including radioactive waste from entering the open atmosphere or other areas where human exposure might result.

This is accomplished by employing dozens to hundreds of various filter testers, including sophisticated machines that produce near-monodisperse test aerosols by vaporization/recondensation processes. For many years, the liquid used in these machines was DOP (dioctyl phthalate). But when DOP was named a possible carcinogen, pressure mounted to replace DOP with a material that would perform like DOP, but would be harmless even in "hot smoke" operations with their attendant high vapor pressures. For several years, DOS (dioctyl sebacate) was used as a DOP replacement. But DOS was not clean to use and did not perform as well as DOP in most cases. And in more recent years, DOS itself became suspect as a possible carcinogen.

Another problem with DOP (or DOS) involved the waste disposal process at Hanford. If contaminated only with DOP/DOS, an expended filter could be disposed of as straight carcinogen or chemical waste. If contaminated only with nuclear waste, it could be disposed of as such. But, since all filters were tested using DOP or DOS, and then were exposed to radiation, the combined disposal problem was very severe and meant that the cost of disposal per filter became prohibitively high.

Thus, DOE was greatly interested in Emery 3004 because it promised filter testing as good as or better than that using DOP or DOS, while at the same time protecting workers from exposure to carcinogens, and greatly reducing the costs of filter disposal. Also very importantly, it allowed all testing machines to be retained without modification, and avoided enormous replacement costs for new equipment.

For eight months, Westinghouse Hanford Company used Emery 3004 as a challenge aerosol for testing of in-place HEPA filters. Their report (Appendix A) is discussed below, and mentions that Emery 3004 does not cause buildup on or plugging of test equipment, as DOP and DOS had.

## 1.3 Eli Lilly and Company

Like Hanford, Lilly is investigating a replacement material for DOP in HEPA filter testing. Lilly made a presentation to the FDA on August 31, 1993, entitled "An Alternative to the Use of Dioctyl Phthalate (DOP) for HEPA Filter Testing in the Pharmaceutical Industry." They shared with FDA the results of studies conducted at ERDEC and other laboratories, and also results of four studies commissioned by Lilly on the comparative performance of DOP versus Emery 3004 (see Appendix B).

The four studies addressed (1) physical property testing; (2) mold growth studies; (3) limited field testing; and (4) an expanded comparative study under field test conditions.

At this writing, Lilly expects to continue its conversion internally to Emery 3004, and to begin to extend this recommendation to the pharmaceutical industry in general. Internal changes include developing a Lilly material specification for Emery 3004, and change of procedures and documents related to HEPA filter testing. These changes are scheduled to be completed in 1Q CY1994.

# 2. EMERY 3004 EVALUATION REPORTS

## 2.1 General

The motivation for the DOP replacement studies and reports generated by the Westinghouse Hanford Company (Appendix A) and Eli Lilly and Company (Appendix B) is very clear. They supplement the many studies carried out by ERDEC over the past five years, and there is general agreement in the conclusions reached.

## 2.2 U.S. Department of Energy (DOE)

In Appendix A, the DOE report compares and directly tabulates the specifications for aerosols of DOP, DOS, and Emery 3004 generated by an ATI, Inc., TDA-100 (Q-127) system. They find that, operationally, Emery 3004 has several advantages over approved performance testing chemicals, including that it is not consudered a carcinogen or suspect carcinigen. Therefore, respiratory protection is not required during testing. Additionally, Emery 3004 does not cause buildup or plugging of the equipment like DOP or DOS.

Tests for DOE by the Westinghouse Hanford Company performed in a practical field demonstration showed that Emery 3004 behaved like the "traditional" aerosols DOP and DOS. Further DOE tests gave particle distribution data, resulting in approval to use Emery 3004 as a challenge aerosol agent for the in-place testing of high-efficiency particulate air (HEPA) filter systems.

Using a cold smoke and a hot smoke machine, Westinghouse Hanford performed in-place penetration testing of HEPA filters from 2 May 1991 through 7 January 1992, during which time 427 HEPA filter systems were challenged. The test results indicated that the readings gathered from filter systems using Emery 3004 were "virtually identical" to those using DOS as the challenge agent.

## 2.3 Eli Lilly and Company

Lilly and other pharmaceutical companies have been seeking a non-carcinogenic replacement material for DOP, which exhibits similar performance in HEPA filter integrity, for several years. In March 1992, an internal task group at Lilly became aware of ERDEC's efforts and its promising candidate material, Emery 3004.

Initial trials at Lilly investigating the relative physical properties of Emery 3004 as compared to DOP produced favorable results. Also, a limited comparative trial of the two materials involving field testing of filters yielded promising results, warranting further studies. In these studies conducted during June and July 1993, an extensive testing program was initiated to compare the performance of Emery 3004 against DOP in the integrity testing of several HEPA filters over a variety of operating conditions. Controlled defects (pinholes) were intentionally made in filters and comparative test data were taken.

In this study, over one hundred comparative data pairs were obtained. The study results produced conclusive evidence that the Emery 3004 compound performed as well or better than DOP in detecting HEPA filter leaks. Lilly performed this research in conjunction with The Joseph Kennedy Company, an Indianapolis-based cleanroom testing contractor and representative of HEPA filter manufacturers, which has been in business since 1960. Kenndey had the required expertise to support this study.

Details of the set-up and three testing series are given in Appendix B. By controlled use of hypodermic needles, pinholes were introduced in the filters which caused the downstream penetrations to vary over a range of 100:1, from about 0.004% to nearly 0.4%. The correlation coefficient between DOP and Emery 3004 performance was found to be 0.995. For comparable upstream concentrations, 95% confidence can be expected that the average concentration reading downstream of a leak as measured using Emery 3004 will be between 1.040 and 1.126 of the reading using DOP.

# 3. CONCLUSIONS

The Westinghouse Hanford Company report (Appendix A), on behalf of DOE, concludes that Emery 3004 is an effective challenge aerosol and is a viable substitute for both DOP and DOS. This relates to different smoke generators, and includes "hot pot" machines that produce smokes by vaporization and recondensation. Further advantages of Emery 3004 are that respiratory protection is not required, that buildup or plugging of test equipment does not occur as it does with DOP and DOS, and that reduced maintenance using Emery 3004 increases the efficiency of company operations.

Lilly (Appendix B) finds that its study data support the effectiveness of Emery 3004 as an acceptable non-carcinogenic replacement for DOP as the challenge aerosol of choice in the integrity testing of HEPA filters. Their results are "consistent" with those reported earlier by ERDEC. Lilly's action plan resulting from this study is that, dependent upon satisfactory results of concurrent testing of the materials' relative fungistatic natures, the company intends to proceed with a recommendation to the industry and appropriate government agencies that Emery 3004 replace DOP in the testing of HEPA filters within the pharmaceutical industry.

Since HEPA filter testing often is conducted using cold smokes generated from Laskin nozzles it is important to remember that, subsequent to the approval by the U.S. Army Surgeon of Emery 3004, this office also gave unlimited approval for testing to a less-viscous PAO liquid, Emery 3002 (Ethylflo 162).[3] Because it is less viscous, Emery 3002 produces cold aerosols with mean particle diameters about half those of Emery 3004. The mass median diameter (MMD) of a cold-sprayed aerosol of Emery 3004 is about 0.7 um, while that of Emery 3002 is about 0.4 um. Because the most penetrating aerosol diameter is about 0.3 um or less, a more rigorous test will result using Emery 3002.

## 4. RECOMMENDATIONS

We recommend that:

- Emery 3004 (Ethylflo 164) be used to replace DOP (or DOS) in all filter test operations;

- Emery 3002 (Ethylflo 162) be assessed for use instead of Emery 3004 in the case of "cold smoke" testing, where smaller MMD of its aerosol can insure even more rigorous filter testing;

- Emery 3002 (Ethylflo 162) <u>not</u> be used in "hot smoke" testing due to its relative volatility; however, Emery 3004 (Ethylflo 164) is perfectly safe in such applications.

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4. Ethyl Corporation, 451 Florida Boulevard, Boca Raton, LA 70801; TEL: (504) 388-7040, FAX: (504) 388-7848.

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APPENDIX A

# Emery 3004 as a Challenge Aerosol: Operational Experience at Westinghouse Hanford Company

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# EMERY 3004* AS A CHALLENGE AEROSOL: OPERATIONAL EXPERIENCE AT WESTINGHOUSE HANFORD COMPANY

D. H. Steffen
C. K. Girres

## ABSTRACT

High Efficiency Particulate Air (HEPA) filter systems are tested periodically by chemicals such as dioctyl phthalate (DOP) and di-2-ethylhexyl sebacate (DOS) to ensure adequate performance. For eight months, Westinghouse Hanford Company used Emery 3004 as a challenge aerosol for in-place HEPA filter system testing.

Operationally, Emery 3004 has several advantages over approved performance testing chemicals, including that it is not considered a carcinogen or suspect carcinogen; therefore, respiratory protection is not required during testing. Additionally, Emery 3004 does not cause buildup on or plugging of the test equipment like DOP or DOS. By reducing the maintenance required on equipment, use of Emery 3004 increases the efficiency of Westinghouse Hanford Company operations.

The concern with using Emery 3004 for in-place testing of HEPA filter systems has been the lack of definitive data on its particle size distribution when generated with a "cold smoke" generator. Quantitative data was not available to show compliance with the particle size distribution requirements of American National Standards Institute, American Society of Mechanical Engineers N510, the standard for in-place testing. To provide comparative

*Emery 3004 is a trademark of the Henkel Corporation.

data between DOP, DOS, and Emery 3004, Westinghouse Hanford Company performed a practical field demonstration, and the results indicated that Emery 3004 behaved like the traditional aerosols. Additional preliminary tests were conducted to obtain particle distribution data, and, as a result of this testing, Westinghouse Hanford Company has received approval from the U.S. Department of Energy-Headquarters, Office of Engineering and Operations Support Defense Programs, and the U.S. Department of Energy, Richland Field Office, to use Emery-3004 as a challenge aerosol agent for the in-place testing of high-efficiency particulate air filter systems via U.S. Department of Energy letter from J. R. Hunter, Assistant Manager for Operations to T. M. Anderson, President of Westinghouse Hanford Company, dated September 24, 1992.

This paper discusses the operational advantages of Emery 3004 and further discusses our test results.

# EMERY 3004 AS A CHALLENGE AEROSOL: OPERATIONAL EXPERIENCE AT WESTINGHOUSE HANFORD COMPANY

Several studies have taken place to look for noncarcinogenic replacement aerosols that behave similarly to dioctyl phthalate (DOP). These studies, which analyze materials that are not carcinogens or suspected carcinogens, are based on efficiency testing.

One of the most recognized studies nationally is *A Study of Candidate Replacement Materials for DOP in Filter-Testing Penetrometer Machines* (Carlon and Guelta 1989). This study was performed by the U.S. Army Armament Chemical Command at Aberdeen Proving Ground, Maryland, and focused on identifying viable candidates to replace DOP as a challenge aerosol. Emery 3004* was chosen as one of the candidate materials.

Both a cold smoke machine and a hot smoke machine similar to the one used in the field comparison testing were used to obtain data. The accepted U.S. Army standard for hot smokes is a geometric mean diameter (GMD) of 0.3 $\mu$m, a geometric standard deviation (GSD) equal to or less than 1.3, and a mass concentration of 100 mg/m$^3$. The experimental procedures were based on these specifications. The hot smoke machine used is known as a Q127, model number TDA-100** manufactured by Air Techniques Incorporated (ATI). The particle size was monitored with a laser aerosol spectrometer.

The approach used to test the candidate materials was to look at the properties of DOP that make it desirable as a test aerosol. DOP is characterized by its low vapor pressure, chemical stability, and insolubility in water. Material properties that were considered include reproducible particle size, size distribution, and smoke concentration (a function of the material density and particle diameter).

The penetrometer used was adjusted to achieve the appropriate particle size and distribution. The GMD was allowed to vary from 0.2$\mu$m to 0.3$\mu$m because recent recommendations for the penetrometer operations include the use of particles smaller than 0.3$\mu$m because they are more effective in penetrating modern filters than are larger particles. To maintain the same vapor pressure, the temperature of the pot was varied.

When Emery 3004 was tested, the GMD was adjustable from 0.2$\mu$m to 0.3$\mu$m, a GSD of 1.23 was obtained, and it had an adequate aerosol yield. The test results showed that Emery 3004 has a high potential as a replacement material for DOP/di-2-ethylhexyl sebacate (DOS). Sample test results for DOP, DOS, and Emery 3004 from the report are shown in Table 1 (Carlon and Guelta 1989).

*Emery 3004 is a trademark of the Henkel Corporation.
**TDA-100 is a trademark of Air Techniques Incorporated.

Table 1. Sample Test Results for COP, DOS and Emery 3004.

| | GMD ($\mu$m) | GSD | Pot Temperature (°C) |
|---|---|---|---|
| DOP | .2581 | 1.542 | 172 |
| DOS | .3017 | 1.161 | 185 |
| Emery 3004 | .2944 | 1.230 | 180 |

The report concludes by recommending Emery 3004 synthetic hydrocarbon as one of two probable replacement materials and recommends further testing to ensure adequate stability and aging characteristics as well as initiating toxicological screening.

A followup of this study was presented at the 21st DOE/NRC Nuclear Air Cleaning Conference: *Safe Replacement Materials for DOP in "Hot Smoke" Aerosol Penetrometer Machines* (Carlon and Guelta 1991). The conclusion of this paper was that a synthetic hydrocarbon can be used to replace DOP directly with minimum impact upon existing hardware and procedures.

In addition to the work described in the 1989 study, further testing was performed to determine the effects of aging and temperature on the stability of Emery 3004. Testing showed that Emery 3004 was sufficiently similar to DOP when subjected to evaporation and recondensation in filter penetrometer testers. The next step in the experimentation process showed that Emery 3004 is thermally stable when subjected to aging tests at elevated temperatures. DOP and Emery 3004 were observed to have similar aging properties.

Westinghouse Hanford Company used Emery 3004 as a challenge aerosol for in-place penetration testing of high-efficiency particulate air (HEPA) filters from May 2, 1991, through January 7, 1992. During this time period, 427 HEPA filter systems were tested. The test results were compared to earlier and subsequent tests that used DOS as the challenge agent on the same filter systems. The results of the tests indicated that the readings gathered from the filter systems using Emery 3004 were virtually identical to those using DOS as the challenge agent.

Additional challenge aerosol comparison tests were conducted at Westinghouse Hanford Company to compare the performance of three challenge aerosols: DOP, DOS, and Emery 3004. The tests were conducted using equipment that is normally used for testing HEPA filter systems in the field at the Hanford Site. Actual field conditions were simulated in the Vent and Balance Laboratory in the 2101-M building in the 200 East Area.

The comparison tests were conducted on January 9, 1992. Three ATI Model TDA-5A* smoke generators were used, one for each aerosol. Each smoke generator was set to the test equipment and aerosol manufacturers' specifications. Three smoke generators were used to facilitate performance of

*TDA-5A is a trademark of Air Techniques Incorporated.

the tests in as short a time span as possible to avoid fluctuations in ambient air pressure and temperature. An ATI Model TDA-2E penetrometer was used to measure smoke penetration through the test filters.

Two 61 x 61 x 30.5 cm (24 x 24 x 12 in.) HEPA filters were used for the tests. The first test series was conducted using an old filter that had been used for several years. The second test series was conducted using a new filter. The third test series was conducted using the old filter with an intentional perforation of 0.16 cm (1/16 in.) diameter located at the filter center.

These three tests were performed to generate data that was as similar as possible to actual field conditions and provide a realistic comparison of the aerosols. All test data, conditions, filters, instrument calibrations and test methods were witnessed and verified by Hanford Site quality control and industrial safety personnel.

The DOP aerosol was tested at the beginning and conclusion of each series of tests to "bracket" the other two aerosols and ensure that the test conditions remained constant. The data indicates that Emery 3004 performs almost identically to DOP and slightly better than DOS. All three aerosols performed within 0.15 percent of each other.

The test data was analyzed only by comparing data within each individual test set (Table 2). Comparisons of data were not made between test sets because the test conditions can change because of factors such as how much air leakage occurs around the filter installation. The purpose of the test was to look at aerosol performance under field conditions, so no effort was made to standardize these conditions.

In the two tests performed on the old filter, it failed in all cases. In addition, for the new filter, the minimum specification was met in all cases. All of the data was within 0.002 percent of each other, which is the standard allowed for the subjectivity in reading the penetrometer. No unusual observations were made that would invalidate the data.

## INTEGRATION OF ANALYSES

Several conclusions can be drawn by looking at our field data and comparing it to laboratory experiments. The U.S. Army literature indicates the critical factor when looking for an equivalent aerosol is the particle distribution. The material chosen must exhibit chemical properties similar to DOP to meet the specifications for both the aerosol and the test machine.

The primary difference between the Hanford Site field test and the tests done at Aberdeen Proving Ground is in the test process itself. The Aberdeen Proving Ground test was designed for efficiency testing. For these tests, an aerosol having a monodispersed particle size is specified. The in-place leak tests done in the field are based on a polydispersed particle size meeting the requirement of American National Standards Institute/American Society of Mechanical Engineers N510 (ANSI/ASME 1989b).

Several similarities between the two tests allow for favorable comparison of the results and the ability to draw accurate conclusions. First, although the smoke generators used were different (as discussed above), they are both manufactured by the same company and operate on the principle of a hot pot to generate the aerosol smoke. The 21st DOE/NRC Air Cleaning Conference Abstract states that the data obtained should be applicable to all hot pot machines (Carlon and Guelta 1991). Secondly, the Aberdeen Proving Ground tests were based on finding replacements based on chemical similarities and toxicological data. The data discussed in this paper supports the conclusion that Emery 3004 is an effective challenge aerosol and viable substitute for both DOP and DOS.

Table 2. Challenge Aerosol Comparison Test Data.

| Test Number 1 (Used HEPA filter) | | | |
|---|---|---|---|
| Aerosol | Range | Penetration | Reading |
| DOP | 1% | 0.6% | 99.4% |
| DOS | 1% | 0.7% | 99.3% |
| Emery 3004 | 1% | 0.6% | 99.4% |
| DOP | 1% | 0.7% | 99.3% |

| Test Number 2 (New HEPA filter) | | | |
|---|---|---|---|
| Aerosol | Range | Penetration | Reading |
| DOP | 1% | 0.002% | 99.998% |
| DOS | 1% | 0.002% | 99.998% |
| Emery 3004 | 1% | 0.003% | 99.997% |
| DOP | 1% | 0.004% | 99.996% |

| Test Number 3 (Used HEPA filter, perforated) | | | |
|---|---|---|---|
| Aerosol | Range | Penetration | Reading |
| DOP | 1% | 0.3% | 99.7% |
| DOS | 1% | 0.2% | 99.8% |
| Emery 3004 | 1% | 0.35% | 99.65% |
| DOP | 1% | 0.3% | 99.7% |

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## APPENDIX B

Eli Lilly and Company, "Comparative Testing of DOP and Emery 3004 in HEPA Filters with Controlled Defects"

Note: This appendix has been renumbered to coincide with the report.

Lilly

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September 17, 1993

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Gentlemen:

Many thanks for the information which you provided my colleague, Ron Wolff, and myself during Lilly's investigation of a replacement for DOP in HEPA filter testing. Lilly made a presentation to the FDA on August 31 entitled "An Alternative to the Use of Dioctyl Phthalate (DOP) for HEPA Filter Testing in the Pharmaceutical Industry". We shared with them various studies related to the search for a DOP replacement in filter testing. Results of studies by the U.S. Army, DOE, Harvard and ATI were referenced. Also, results of four studies commissioned by Lilly on the comparative performance of DOP versus Emery 3004 were shared. Those were (1) physical property testing; (2) mold growth studies; (3) limited field testing; and (4) an expanded comparative study under field test conditions. As one way of sharing information with others doing work in this field, I am forwarding to you a copy of the report from our expanded study, "Comparative Testing of DOP and Emery 3004 in HEPA Filters with Controlled Defects".

Bob Sorensen of the CDER Division of Manufacturing and Product Quality was our primary contact for the FDA presentation. Mr. Sorensen and his colleagues reviewed the data, conclusions and recommendations from our presentation. They appear supportive, up to this point, of Lilly continuing its conversion internally to Emery 3004 and our beginning to roll out this change to the pharmaceutical industry in general. The internal changes involve such things as developing a Lilly material specification for Emery 3004 and proper change control of appropriate procedures and documents related to HEPA filter testing. Our target for completing this change within Lilly, if all continues to go well, is the first quarter of 1994. The roll-out to industry will involve presentations to various industry groups (i.e. PDA, PMA, ISPE) and an article or two in pharmaceutical industry technical publications. Jeff Marshall (co-author of the attached report) and I are scheduled to present on this subject at the Raleigh (November, 1993) and Tampa (February, 1994) seminars of ISPE.

Again, thank you for your help. I hope that this update on our progress and the attached report prove informative to you.

Sincerely,

Donald R Moore 9/20

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# Comparative Testing of DOP and Emery 3004 in HEPA Filters with Controlled Defects

August 27, 1993

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## EXECUTIVE SUMMARY

Dioctyl phthalate (DOP) has been the standard material for testing the performance of High Efficiency Particulate Air (HEPA) filters in military and industrial applications for several decades. Concern about the safety (carcinogenicity) of DOP has been increasing in recent years, and in 1985 it was listed as a suspected carcinogen by the U.S. Surgeon General. The integrity leak testing of installed HEPA filters involves challenging the filters with a "cold" (ambient) DOP aerosol with a range of particle sizes having an average diameter of about 0.7 micron. For several years, the pharmaceutical industry has been seeking a non-carcinogenic replacement material for DOP which exhibits similar performance in HEPA filter integrity testing. An internal task group, commissioned by Lilly to investigate potential replacement materials, became aware in March 1992 of other similar efforts to find a substitute for DOP. The most promising candidate from these efforts was the synthetic hydrocarbon Emery 3004. Initial Lilly studies investigating the relative physical properties of Emery 3004 as compared to DOP produced favorable results. Also, a limited comparative trial of the two materials involving field testing of filters yielded promising results, warranting further studies.

In this study, conducted during June and July 1993, an extensive testing program was initiated to compare the performance of Emery 3004 against DOP in the integrity testing of several HEPA filters over a variety of operating conditions. Controlled defects (pinholes) were intentionally made in the filters and comparative test data taken.

Over one hundred comparative data pairs were taken in this study. The results of the study produced conclusive evidence that the Emery 3004 compound performed as well or better than DOP in detecting HEPA filter leaks.

The intended action plan from this study is that Lilly will propose to the appropriate agencies that the non-carcinogenic Emery 3004 replace DOP as the preferred challenge aerosol for HEPA filter testing in the pharmaceutical industry.

# COMPARATIVE TESTING OF DOP AND EMERY 3004 IN HEPA FILTERS WITH CONTROLLED DEFECTS

## I. Background

Concern within Eli Lilly and Company regarding the continued use of the suspected carcinogen dioctylphthalate (DOP) for HEPA filter testing led to the formation of an internal multi-disciplined task group in March 1992 with the mission of seeking an acceptable replacement material. In its investigation for a non-carcinogenic alternative to DOP, the task group learned of research already underway to find a replacement by others such as Carlon (3, 7), Crosby (5) and First, et al (6). The most commonly recommended replacement material from these studies was a well-defined synthetic hydrocarbon, a 4-centistoke poly-alpha olefin (PAO) manufactured by the Henkel Corporation called Emery 3004. Lilly commissioned internal work by Wolff (1) to further study the physical properties of Emery 3004 and by Ulmer (2) to do a limited comparative study of DOP in the actual field testing of HEPA filters. The results of these studies were encouraging enough for Lilly to commission a more expanded comparative study of the effectiveness of Emery 3004 as a replacement for DOP in HEPA filter integrity testing.

In June 1993, Lilly began an extensive series of tests to compare the effectiveness of the two challenge aerosols in a cooperative effort with the Joseph Kennedy Company. The Joseph Kennedy Company is an Indianapolis-based cleanroom testing contractor and HEPA filter manufacturers representative which has been in existence since 1960. The Joseph Kennedy Company had the required expertise in filter testing and the equipment needed to support this study. Testing and data collection was performed during June and July of 1993 by J.G. Marshall and M.A. Kennedy of The Joseph Kennedy Company and D.R. Moore of Eli Lilly and Company.

## II. Purpose:

The purpose of these tests was to evaluate the comparative performance of Emery 3004 as a potential replacement challenge aerosol for DOP in the integrity testing of HEPA filters. These tests were intended to eliminate variations in test equipment and techniques which might bias the direct comparison of DOP and Emery 3004. The intent was that this comparison of performance be done under conditions which as nearly as possible duplicated those that exist in actual field test applications. If it did prove to perform comparably to DOP, Lilly would propose to pursue with the appropriate agencies the substitution of Emery 3004 for all HEPA filter testing within Lilly and the remainder of the pharmaceutical industry.

## III. Procedure Overview:

Tests were performed using the Joseph Kennedy Company's test unit for demonstrating and evaluating air filters. This unit is capable of producing an aerosol-laden air stream of up to 2,000 cfm at 2.5 inch W.G. (Appendix II, Exhibit A). An upstream HEPA prefilter was added for these tests in order to reduce the particulate burden in the air stream prior to the injection of the challenge aerosol. For the sample filters, a random group of three HEPA filters from two different manufacturers was selected as the control group. Two of these were the fluid-seal design with separatorless media packs and one was the gasket seal type with aluminum separators. Prior to the start of testing, test filters were installed into the test rig and scan tested according to I.E.S. RP-CC-006 Section 5.1. If necessary, they were repaired in order to establish satisfactory filter seals and media packs. A series of controlled defects was then introduced to each filter using a test grid (template) with ten equally spaced pinholes using different gages of hypodermic needles (Appendix II, Exhibit B). Defect sizes ranged from .012 inch to .075 inch. Both photometers used in these tests were ATI Model TDA-2E units and were factory calibrated using DOP as the internal light reference standard, which is customary for the filter industry. Upstream challenge aerosol concentrations were recorded prior to each run, and the photometer was then calibrated to 100% of the upstream concentration. Downstream leak penetration readings were then recorded as a percent of the upstream concentration. HEPA filter test standards define a leak requiring repair to be any reading of .01% or more of the upstream concentration.

Three different series of tests were run as follows:

A. Testing Series I (Test Procedure I, Appendix I) involved eight test runs (Tests 1 through 8) in which multiple test equipment sets (two photometers and two generators) were varied while testing the leakage of both DOP and Emery 3004 through a filter in which controlled defects had been introduced. The airflow velocities were held fairly constant (within 10% of the target of 342 fpm). The photometers and generators were of identical manufacturer and model. In this test, the photometer probe was moved between comparative data runs as equipment and challenge aerosols were changed. The purpose of this series of tests was to identify any variations in test equipment or methods that might introduce a bias which could interfere with the direct comparison between the performance of DOP and Emery 3004.

B. Testing Series II (Test Procedure II, Appendix I) involved twelve test runs (Tests 9A through 12B, and 17A through 18B). The intent of these tests was to reduce or eliminate any bias in test equipment or methods which might interfere with the direct comparison between the performance of DOP and Emery 3004. Comparative data for each pinhole was taken at each velocity for DOP and Emery 3004 without moving the photometer probe. Only one photometer (Photometer A) was used. Generator A remained filled with DOP throughout the test, and Generator B remained filled with Emery 3004. Tests were run over a wide range of air velocities from 59 FPM to 514 FPM.

C. Testing Series III (Test Procedure III, Appendix I) involved eight test runs (Tests 13A through 16B). This test was intended to determine if any bias may have been introduced due to the sequence in which DOP and Emery 3004 data was taken (i.e., Does the sequence of taking the data make a difference?). This might occur if one of the materials were to act as a solvent for the other. Data was taken by holding all other parameters constant and taking readings while switching between DOP and Emery 3004 in various sequences. Only two penetrations were studied, near opposite extremes in terms of size of the defect.

Data collected from each series of tests is recorded in Appendix III.

## IV. Results:

General Observations:

- In all three series of tests, there was only a general correlation between the size of the controlled defect (pinhole) and the relative amount of leakage as measured by percent of upstream concentration. The manipulation of a given hypodermic needle and the profile of its point had a dramatic effect on the corresponding leakage. The important fact is that the controlled defects did introduce leakages which allowed comparison of DOP and Emery 3004 over a wide range (data points over 3 logarithmic scales) of downstream concentrations (from about .004% to nearly .4%). It is also interesting to note that some of the pinholes introduced would have passed a scan test (less than .01% leakage).

- In the first test series, we did observe that the factor which could potentially introduce the greatest variation in test results was the moving of the photometer probe between comparative readings (i.e., DOP vs. Emery at the same leak point at the same velocity with the same photometer). This is not obvious from a review of the data alone. Every effort was made to reposition the probe and its stand in exactly the same relative position each time by use of a ruler and T-square. However, the exact distance and orientation of the probe relative to the pinhole and leakstream is so sensitive that extremely slight variations in the position would introduce differences in the readings that were several times the magnitude of the relative expected differences we were trying to determine between DOP and Emery 3004. This led to fixing the location of the photometer probe during comparative readings in subsequent test series.

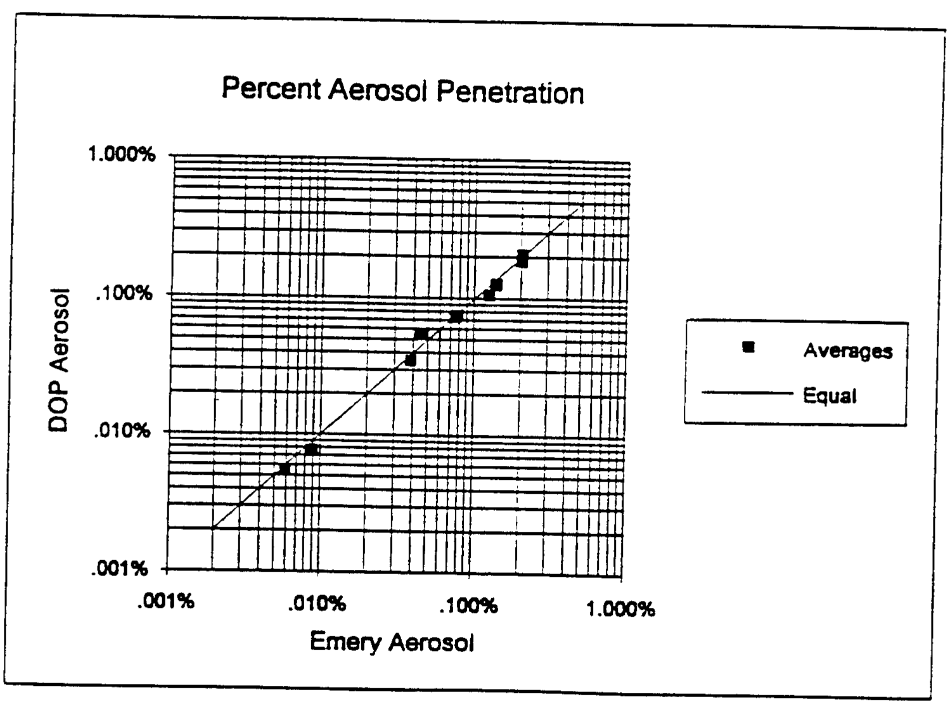
- Relative photometric readings for Emery 3004 of upstream concentrations generally read higher than comparable readings for DOP. This is consistent with findings from previous work by Wolff (1), Ulmer (2), Carlon (3, 4, 7), Crosby (5) and First, et al (6).

The results of the first series of tests (Appendix III; Testing Series I) were studied to determine which variations in the test equipment or methods might introduce a bias into the comparative testing of DOP and Emery 3004. This analysis found that the effect of different generating and scanning equipment or conditions in the various runs was insignificant in the comparison of DOP and Emery 3004. A review of the data from the first series (plotted in Graph A of this section) indicates that any effect on the data which might have been introduced by moving the photometer probe between comparative readings equally affected both sets of readings on the average and a strong case can still be made from this data that Emery 3004 performs as well or better than DOP in detecting leaks.

*In reviewing Graphs A, B, C, D, and Table A, it is important to note that if DOP and Emery 3004 were exactly the same material and tested by our methods, due to statistical variation one would expect an equal number of data points scattered on both sides of the 1:1 plot line (DOP reading = Emery 3004 reading). A scattering of data points shifted to one side of this line or the other indicates a generally higher average reading for that material versus the other.*

GRAPH A: TESTING SERIES I DATA

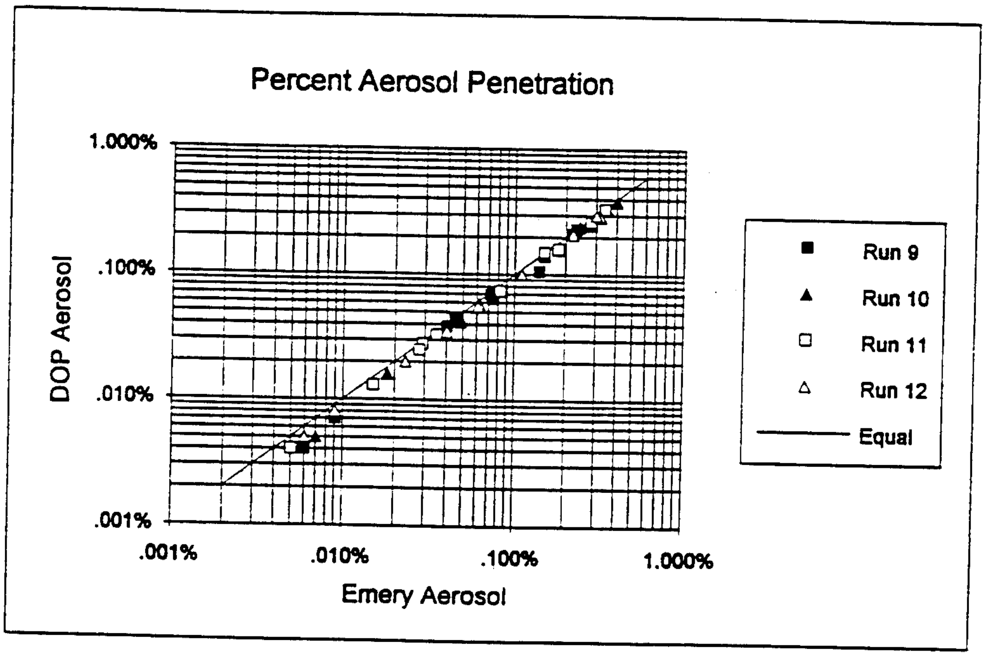
(Runs 1-8)



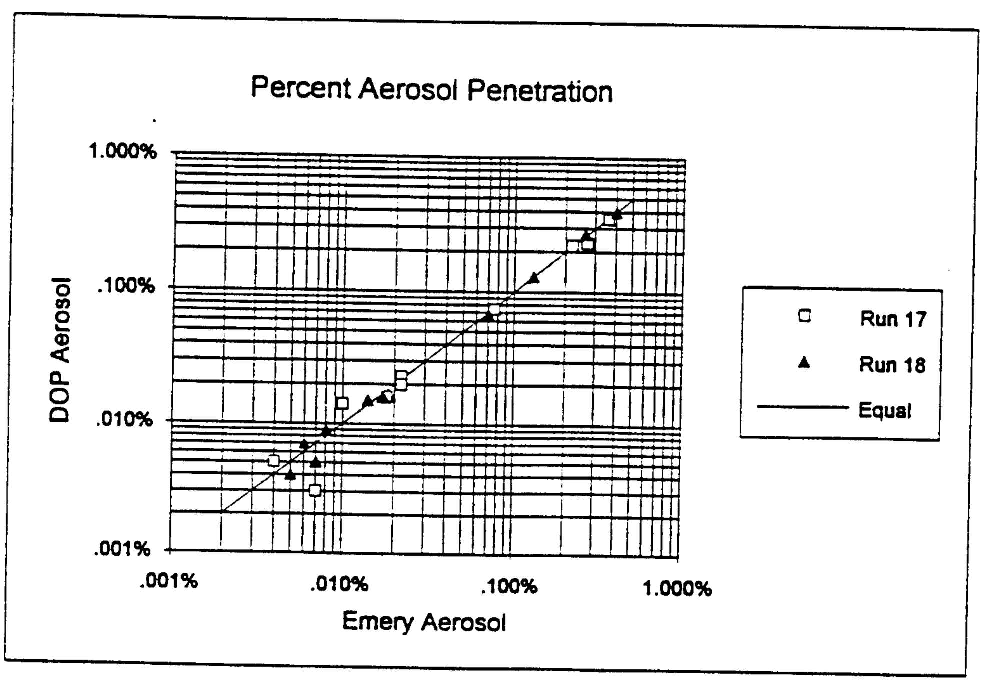
In the second series of tests (Appendix III; Testing Series II) the position of the photometer probe remained fixed while comparative readings were taken at each pinhole. In this test, sixty pairs of data points were generated on three different filters. The data represents variations of air velocities, size of pinhole leaks, and filters. Each data pair (i.e., 9A #1/9B #1, or 17A #5/17B #5) had the following held constant for those two points: air velocity, pinhole size, filter, air temperature, air relative humidity, photometer and, most importantly, photometer probe position. This set of data provides the most direct comparison between the performance of DOP and Emery 3004 in filter integrity scan testing. The results of the test (Graph B) indicate a very strong case that Emery 3004 performs as well or better than DOP in detecting filter leaks.

## GRAPH B: TESTING SERIES II DATA

### Graph B.1 (Runs 9-12)

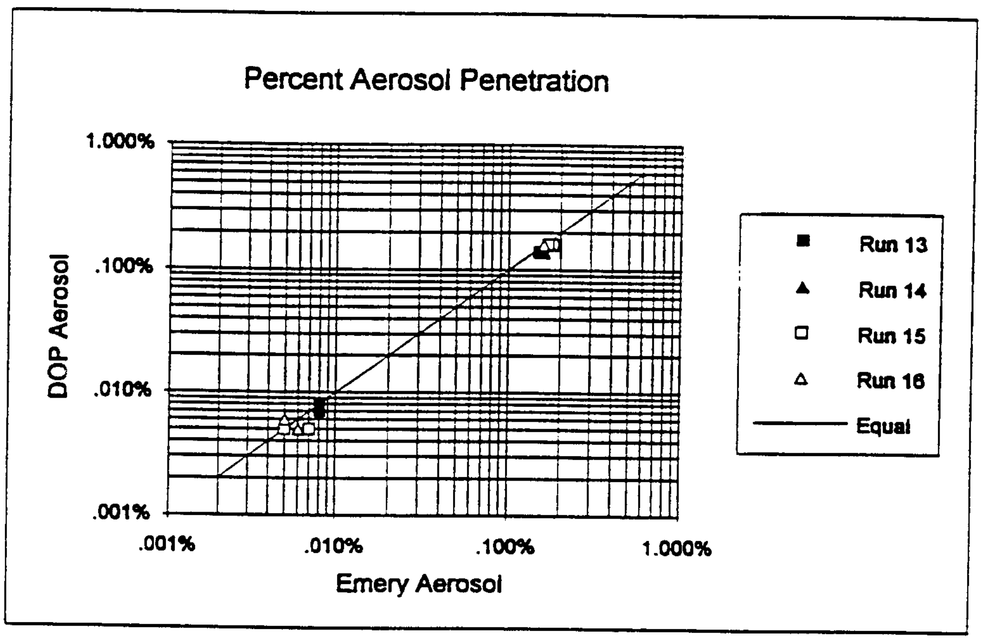



### Graph B.2 (Runs 17 & 18)



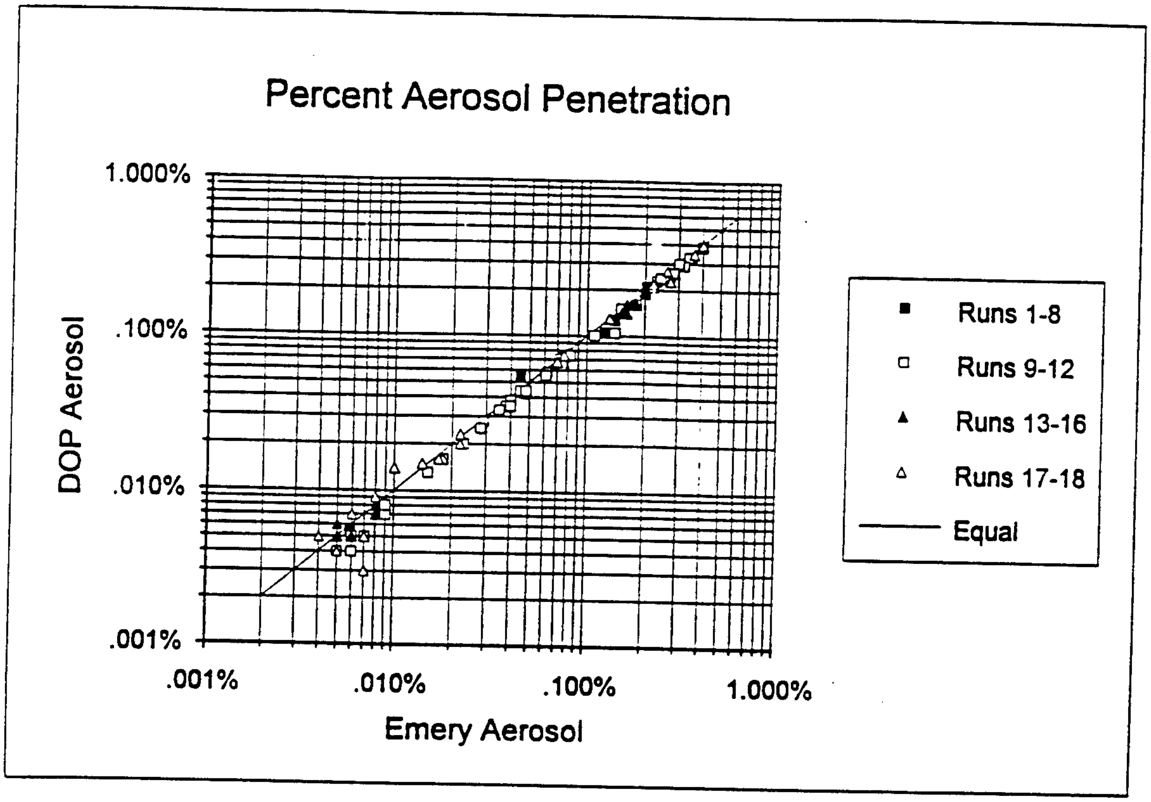
In the third series of tests (Appendix III, Testing Series III) the results were analyzed to determine if the sequence in which the DOP and Emery 3004 data was taken introduced any bias in the results. In reviewing the results of this test, there is no indication that the order in which DOP and Emery 3004 data was collected in any way affected the results. However, the data (Graph C) continues to support the trend of results from the first two series of tests in verifying that Emery 3004 performed as well or better in detecting filter leaks.

GRAPH C: TESTING SERIES III DATA




In Graph D and Table A, all 116 data pairs from the three testing series are plotted on the same graph and analyzed from the perspective of being over/under the 1:1 line.

## GRAPH D: ALL TEST RUNS COMBINED




## TABLE A: ALL TEST RUNS COMBINED

### COMPARATIVE SCAN TEST RESULTS

(Penetration readings for DOP Greater Than/equal to/Less Than those for Emery 3004)

| Testing Series | DOP G.T. Emery 3004 | DOP equals Emery 3004 | DOP L.T. Emery 3004 | TOTAL |
|---|---|---|---|---|
| I | 6 (15%) | 11 (27.5%) | 23 (57.5%) | 40 |
| II | 7 (11.7%) | 7 (11.7%) | 40 (76.6%) | 60 |
| III | 1 (6.3%) | 4 (25%) | 11 (68.7%) | 16 |
| TOTAL | 14 (12%) | 22 (19%) | 80 (69%) | 116 |

An in-depth analysis of this data by Lilly statistician, John R. Murphy, Ph.D., is included in Appendix III. Paraphrased conclusions from Dr. Murphy's report "Statistical Analysis of the HEPA Filter Challenge Aerosol Comparison Testing Performed by Eli Lilly and the Joseph Kennedy Company" are as follows:

1) When results of data pairs (DOP vs. Emery 3004) are plotted on a log-log scale, the correlation coefficient of the plot is .995.

2) For comparable upstream concentrations, we can expect with 95% confidence that the average concentration reading downstream of a leak as measured using Emery 3004 will be between 1.040 and 1.126 of the reading using DOP.

*The results of each separate testing series as well as the total body of data prove that regardless of the variation in test equipment, air velocities, air conditions, filter type and test methodology, Emery 3004 consistently performs as well or better than DOP in HEPA filter integrity testing.*

V. Conclusions:

The data collected in this study supports the effectiveness of Emery 3004 as an acceptable non-carcinogenic replacement for DOP as the challenge aerosol of choice in the integrity testing of HEPA filters. Our test results are consistent with those reported previously by Carlon (3, 4, 7) and Ulmer (2) in their investigations. The action plan from this study is that, dependent upon satisfactory results of concurrent testing of their relative fungistatic natures, Lilly intends to proceed with a recommendation to the industry and appropriate government agencies that Emery 3004 replace DOP in the testing of HEPA filters within the pharmaceutical industry.

## VI. References

1. Wolff, R.K., K.H. Carlson and R.L. Tielking, December 1992, *Particle Sizing of DOP, Mineral Oil, and Emery 3004 Aerosols,* Eli Lilly and Company, Indianapolis, Indiana

2. Ulmer, J.W., December 1992, *HEPA Filter Testing Report for Emery 3004: Proposed DOP Substitute,* Eli Lilly and Company, Indianapolis, Indiana

3. Carlon, H.R. and M.A. Guelta, March 1989, *A Study of Candidate Replacement Materials for DOP in Filter-Testing Penetrometer Machines*; U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, Maryland

4. Carlon, H.R. and M.A. Guelta, June 1992, *Implementation of DOP Replacement with Selected Materials in Mask and Filter Testing Penetrometer Machines: Final Report,* U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, Maryland

5. Crosby, D.W., August 1991, *Concentrations Produced by a Laskin Nozzle Generator: A Comparison of Substitute Materials and DOP,* Proceedings of the 21st DOE/NRC Nuclear Air Cleaning Conference, San Diego, California, Vol. 1, Page 109-115

6. First, M.W., S.N. Rudnick and Xiaowei Yan, August 1991, *Characteristics of Laskin Nozzle Generated Aerosols,* Proceedings of 21st DOE/NRC Nuclear Air Cleaning Conference, San Diego, California, Vol. 1, Page 116-125

7. Carlon, H.R. and M.A. Guelta, August 1991, *Safe Replacement Materials for DOP in "Hot Smoke" Aerosol Penetrometer Machines,* Proceedings of the 21st DOE/NRC Nuclear Air Cleaning Conference, San Diego, California, Vol. 1, Page 126-138

# APPENDIX I

| | |
|---|---|
| Test Procedure I | Comparative Testing of Filter Test Equipment |
| Test Procedure II | Comparison of DOP and Emery 3004 in HEPA Filter Integrity Testing |
| Test Procedure III | Sequence of Testing Comparison |
| Test Procedure IV | Generator Cleaning |

## TEST PROCEDURE I: Comparative Testing of Filter Test Equipment

Purpose of Test: Comparison testing of test equipment in order to identify any possible significant differences in the test equipment or technique which might bias the comparative testing between DOP and Emery 3004.

Procedure:

1) Fill Generator A with DOP, and fill Generator B with Emery 3004.

2) Prepare template as shown from Appendix II, Exhibit B layout, with each size penetration tagged on template referencing appropriate needle size. The penetrations are not sequential from larger to smaller sizes necessarily, as a large number of penetrations in the same area may contribute enough aerosol concentration to effect smaller magnitude leaks.

3) Insert HEPA filter into holding frame fixture. Secure holding frame with filter to test unit.

4) Establish initial velocity reading through the filter at average of about 342 fpm (per Mil. Std. 282). Measure and record same.

5) Measure and record relative humidity and temperature.

6) Inject DOP challenge aerosol through injection port using Generator A, record upstream concentration using internal light reference feature and calibrate photometer to 100% per IES-RP-CC-006-84 Par. 5.1. Scan entire perimeter of filter-frame to holding-frame seal to insure no bypass leaks are occurring. Repair leaks above acceptable level (.01% of upstream concentration).

7) Penetrate first HEPA filter using template prepared in Step 2.

8) Recalibrate photometers prior to each reading.

9) Move probe and stand for each leak location. Measure and record downstream penetration at end of ten (10) locations.

10) Test HEPA filter for eight (8) test sequences of ten (10) downstream penetration values in the following order:

#1 DOP/Generator A/Photometer A
#2 DOP/Generator A/Photometer B
#3 DOP/Generator B/Photometer B
#4 DOP/Generator B/Photometer A

Flush aerosol generators following Test Procedure IV.

#5 Emery/Generator A/Photometer A
#6 Emery/Generator A/Photometer B
#7 Emery/Generator B/Photometer B
#8 Emery/Generator B/Photometer A

Note: Throughout all tests, an injection hose was dedicated to each material, i.e., a "DOP hose" and an "Emery 3004 hose" which saw only those materials during the test.

(This procedure used for Tests 1, 2, 3, 4, 5, 6, 7 and 8.)

## TEST PROCEDURE II: Comparison of DOP and Emery 3004 in HEPA Filter Integrity Testing

Purpose of Test: Compare the performance of DOP versus Emery 3004 in HEPA filter integrity testing; eliminating as much as possible any differences in testing equipment or techniques which might bias the testing.

Procedure:

1) Fill Generator A with DOP, and fill Generator B with Emery 3004.

2) Prepare template as shown from Appendix II, Exhibit B layout, with each size penetration tagged on template referencing appropriate needle size. The penetrations are not sequential from larger to smaller sizes necessarily, as a large number of penetrations in the same area may contribute enough aerosol concentration to effect smaller magnitude leaks.

3) Insert HEPA filter into holding frame fixture. Secure holding frame with filter to test unit.

4) Establish initial velocity reading through the filter at average of about 342 fpm (per Mil. Std. 282). Measure and record same.

5) Measure and record relative humidity and temperature.

6) Inject DOP challenge aerosol through injection port using Generator A, record upstream concentration using internal light reference feature and calibrate photometer to 100% per IES-RP-CC-006-84 Par. 5.1. Scan entire perimeter of filter-frame to holding-frame seal to insure no bypass leaks are occurring. Repair any leaks above acceptable level (.01% of upstream concentration).

7) Penetrate first HEPA filter using template prepared in Step 2.

8) Secure photometer probe using stand and position one inch downstream of filter face at leak position #1. Measure and record leak penetration value.

9) Switch to Generator B and inject Emery 3004. Recalibrate photometer to 100% of upstream concentration. Measure and record leak penetration value.

10) Reposition photometer probe to one inch downstream of filter face at leak position #2. Recalibrate photometer to 100% of upstream concentration. Measure and record penetration value for Emery 3004.

11) Switch to Generator A and inject DOP. Recalibrate photometer to 100% of upstream concentration. Measure and record leak penetration value.

12) Reposition photometer probe to one inch downstream of filter face at leak position #3. Repeat steps 8 through 11, alternating generators as required to maintain the position of the probe while data is gathered on both DOP and Emery 3004 at each location, until all penetrations have been scanned.

13) Repeat steps 4 through 11 at a different air velocity (eliminating step 7 and the perimeter scan in step 6).

Note: Throughout all tests, an injection hose was dedicated to each material, i.e., a "DOP hose" and an "Emery 3004 hose" which saw only those materials during the test.

(This procedure used for Tests 9A, 9B, 10A, 10B, 11A, 11B, 12A, 12B, 17A, 17B, 18A, 18B.)

## TEST PROCEDURE III: Emery vs. DOP Sequence of Testing Comparison

Purpose: To verify that Emery and DOP do not act as solvents to each other, thus affecting magnitude of leak readings through photometer. This is to determine if the sequence in which DOP and Emery 3004 are tested introduces a bias in the results.

Procedure:

1) Select two (2) pinholes for test purposes, with one being a larger penetration and the other being a smaller pinhole.

2) Set photometer probe at location of leak, supported by a stand, and begin testing based on sequence of four (4) total readings at each pinhole for each series of tests.

3) Select one challenge aerosol of interest, calibrate photometer to 100% of upstream concentration, and record reading.

4) Continue challenging filter for five (5) minute duration following reading to deposit residual challenge.

5) Switch generator to alternate challenge aerosol, and repeat above procedure. Record results for both pinhole locations, together with magnitude of reading.

6) Repeat test at a second velocity to determine any effect which changing airflow might have on the test.

Note: Throughout all tests, an injection hose was dedicated to each material, i.e., a "DOP hose" and an "Emery 3004 hose" which saw only those materials during the test.

(This procedure used for tests 13A, 13B, 14A, 14B, 15A, 15B, 16A and 16B.)

## TEST PROCEDURE IV: Generator Cleaning

Purpose of test: The following procedure is to be followed when changing the aerosol generator from DOP to Emery 3004 use.

Procedure:

1) Drain the machine of DOP

2) Flush with Denatured Alcohol

3) Drain the machine of Alcohol

4) Run the generator with compressed air to dry the Alcohol

5) Flush with Emery 3004

6) Drain this initial flush quantity of Emery 3004

7) Fill with Emery 3004 to run the machine

Follow the same procedure when changing from Emery 3004 to DOP use, switching the words "DOP" and "Emery 3004".

# Appendix II

1. Exhibit A - Test Equipment Set-up Description
2. Exhibit B - Needles and Template Used to Make the Controlled Defects
3. Exhibit C - Equipment List of Test Equipment
4. Exhibit D - Calibration Reports of Test Equipment

## EXHIBIT A

## Test Equipment Set-up Description

The test unit consisted of a damper (1), HEPA prefilter (2), blower (3) and mixing/sampling chamber (4) capable of producing an air-aerosol mixture of 2,000 C.F.M. at 2.5" W.G. The test HEPA filter (5) was loaded into a test duct which was then clamped to the discharge of the unit using a metal frame. Each HEPA filter was overlaid with a template for the purpose of locating the pinholes. These pinholes were made with ten different diameters of hypodermic needles in a predetermined pattern. DOP and Emery 3004 were introduced via the challenge aerosol injection port (6). The downstream sampling probe (7) was mounted to a ring stand with clamp and cast iron base which stood on a resilient mounting pad for purposes of vibration isolation. The air upstream of the test HEPA filter was evaluated prior to the test for particle loading and was found to contain an average of approximately 3,500 particles (0.5 microns) per cubic foot.

Airflow volumes were changed by adjusting the inlet damper (1).

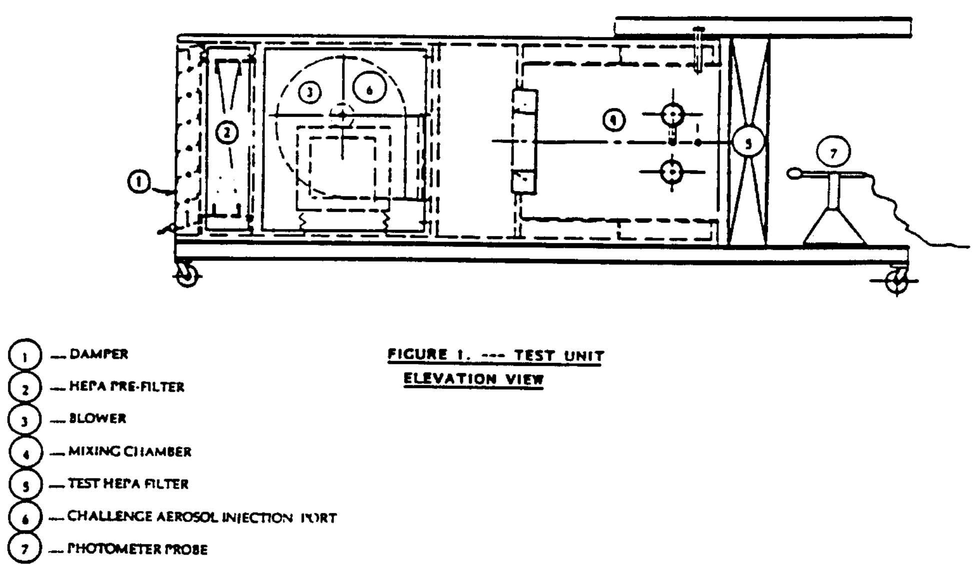
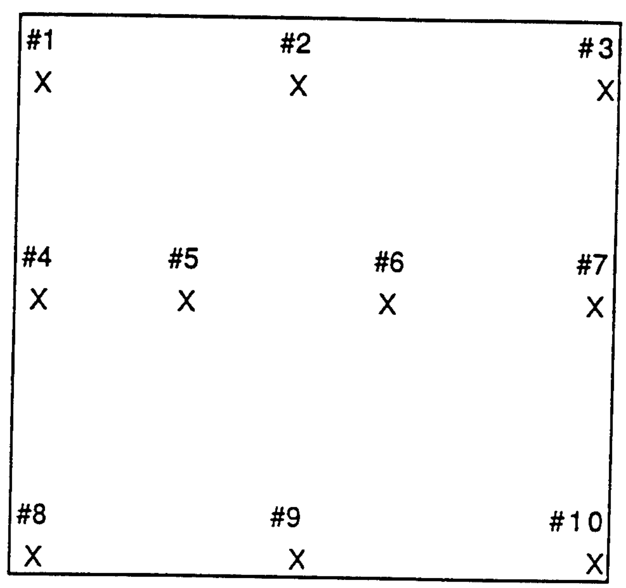



FIGURE 1. --- TEST UNIT ELEVATION VIEW

## EXHIBIT B

| HYPODERMIC NEEDLE GAGE | LEAK POINT | BLUNT END COLOR | NOMINAL SIZE | SIZE AS MEAS. AT JKC |
|---|---|---|---|---|
| 16 | #9 | Silver | .0625" | .075" |
| 18 | #7 | Pink | .050" | .051" |
| 20 | #10 | Yellow | .0375" | .035" |
| 21 | #4 | Green | .0343" | .032" |
| 22 | #2 | Gray | .0312" | .028" |
| 23 | #6 | Turquoise | .028" | .024" |
| 25 | #8 | Blue | .0218" | .020" |
| 26 | #3 | Brown | .0187" | .018" |
| 27 | #5 | Transparent | .017" | .018" |
| 30 | #1 | Flesh | .0125" | .012" |

## FIGURE 2
## FILTER LEAK POINT TEMPLATE



# EXHIBIT C

## Equipment List

1) Photometers:
Air Techniques Model TDA-2E forward light scattering photometer with internal light reference feature - digital readout

Photometer A - Serial #9927
Photometer B - Serial #9246

2) Aerosol Generators:
Air Techniques Model TDA-4A aerosol generators, with eight (8) Laskin nozzles available

Generator A - Serial #9213
Generator B - Serial #9894

3) Air Flow Measurements:
Shortridge Instruments ADM-860 Airdata Multimeter, electronic micromanometer with Velgrid attachment

Serial #M93068

4) Particle Counter:
Met One Model 205 Laser Particle Counter with humidity/temperature probe attachment - Model 085A

Serial #93510

5) HEPA Filters:
Filter #1 - Flanders Blue Gel seal filter with separatorless media pack, 24 in. x 24 in. x 12 in., Model T-007-8-05-05-IU, Serial #743880

Filter #2 - AAF Gasket seal filter with aluminum separators, 24 in. x 24 in. x 12 in., Model 1431915-539, Serial #41456402

Filter #3 - Flanders Blue Gel seal filter with separatorless media pack, 21 1/2 in. x 45 1/2 in. x 3 in., Model 0-007-2-19-06-SU, Serial #L228755

EXHIBIT D

# Calibration Reports of Test Equipment

UNIT #1
CAL #3

**AIR TECHNIQUES**
Division of Hamilton Assoc., Inc.
11403 Cronridge Drive • Owings Mills, MD 21117-2247 USA • Tel 410.363.9696 • Fax 410.363.9695

# NIST TRACEABLE CERTIFICATION & CALIBRATION REPORT
(National Institute of Standards & Technology)

Customer: Joseph Kennedy Co., Inc — ID #:

P.O. #: MK — Job #: 92 1801

Model #: TDA-2E — Serial #: 9246

| CALIBRATION EQUIPMENT USED | Serial # | NIST CAL DATE | CAL DUE DATE |
|---|---|---|---|
| ☒ Keithly Picoamp Source | 41428A | 16 Dec 91 | 16 Dec 92 |
| ☐ Keithly Picoamp Source | 69457 | | |
| ☒ Fluke DMM | 2076149 | 28 Feb 92 | 28 Feb 93 |
| ☐ Fluke DMM | 4955031 | | |
| ☒ Sprague Gas Meter | 2900160A | 18 Dec 91 | 18 Dec 92 |
| ☐ | | | |

| CALIBRATION NOTES | | AS FOUND | AS LEFT | MFG TOLERANCES |
|---|---|---|---|---|
| Straylight | (NA) | .030 % | .021 % | Not Applicable |
| INTernal REF. | (*) | 9.2 % | 10.0 % | 10.0% ± 1.0% |
| .01% | (A) | $1.00 \times 10^{-9}$ | $1.00 \times 10^{-9}$ | $1.0 \pm 0.1 \times 10^{-9}$ amperes |
| .1% | (A) | $1.00 \times 10^{-8}$ | $1.00 \times 10^{-8}$ | $1.0 \pm 0.1 \times 10^{-8}$ amperes |
| 1.% | (A) | $1.00 \times 10^{-7}$ | $1.00 \times 10^{-7}$ | $1.0 \pm 0.1 \times 10^{-7}$ amperes |
| 10.% | (A) | $1.00 \times 10^{-6}$ | $1.00 \times 10^{-6}$ | $1.0 \pm 0.1 \times 10^{-6}$ amperes |
| 100.% | (A) | $1.00 \times 10^{-5}$ | $1.00 \times 10^{-5}$ | $1.0 \pm 0.1 \times 10^{-5}$ amperes |
| Sample Flow | (A) | 31.0 SLPM | 28.3 SLPM | 28.3 ± 2.8 SLPM |

Scanning Probe body broken

* Internal Ref refers to a known concentration level and has no effect upon instrument operation.

( A ) In tolerance when received ( B ) Out of tolerance when received ( C ) Inoperable

( D ) As found results not applicable - new instrument ☐ TOLERANCE NOT APPLICABLE TO THIS INSTRUMENT.

## MAINTENANCE PERFORMED

| | | | |
|---|---|---|---|
| ☒ Rework Scattering Chamber | ☐ Replace Standard Lamp | ☒ Test Absolute Filter | ☒ Align Optics |
| ☐ Replace Smoke Chamber | ☒ Test Electrical Connections | ☐ Replace Gaskets | ☒ Replace Probe Body |
| ☒ Clean Sampling System | ☒ Perform Voltage Measurements | ☒ Tighten Loose Hardware | ☒ Install flow control valve |
| ☒ Replace Cell Lamp | ☒ Flush Vacuum Pump | ☒ Leak Check | ☒ Final Test |
| ☒ Adjust Internal Reference | ☒ Test Scanning Probe | ☐ Align Amplifier | ☒ Final Inspection TM |

CALIBRATION PROCEDURES USED — TEMP: 23.0 °C — R.H.: 47 %

| | | | |
|---|---|---|---|
| ☐ ATI-0001 | ☐ ATI-0004 | ☐ ATI-0006 | ☒ ATI-0036A |
| ☐ ATI-0003 | ☐ ATI-0005 | ☐ ATI-0007 | ☐ |

CALIBRATED BY: Gary McCurdy — B29 — DATE: 30 Sept 92

QA REVIEW: — REVISED: 4/21 RWC

UNIT #2
CAL. #1

**AIR TECHNIQUES**
Division of Hamilton Assoc., Inc.
11403 Cronridge Drive • Owings Mills, MD 21117-2247 USA • Tel 410.363.9696 • Fax 410.363.9695

# NIST TRACEABLE CERTIFICATION & CALIBRATION REPORT
(National Institute of Standards & Technology)

Customer: Joseph Kennedy Co., Inc.    ID #: 

P.O. #: 104AT1    Job #: 930338

Model #: TDA-2E    Serial #: 9927

| CALIBRATION EQUIPMENT USED | Serial # | NIST CAL DATE | CAL DUE DATE |
|---|---|---|---|
| ☐ Keithly Picoamp Source | 41428A | | |
| ☒ Keithly Picoamp Source | 69457 | 5 Aug 92 | 5 Aug 93 |
| ☐ Fluke DMM | 2076149 | | |
| ☒ Fluke DMM | 4955031 | 2 Oct 92 | 2 Oct 93 |
| ☒ Sprague Gas Meter | 2900160A | 18 Dec 91 | 18 Dec 92 |
| ☐ | | | |

| CALIBRATION NOTES | | AS FOUND | AS LEFT | MFG TOLERANCES |
|---|---|---|---|---|
| Straylight | ( NA ) | | .013% | Not Applicable |
| INTernal REF. | ( * ) | | 10.0% | 10.0% ± 1.0% |
| .01% | ( D ) | | $1.00 \times 10^{-9}$ | $1.0 \pm 0.1 \times 10^{-9}$ amperes |
| .1% | ( D ) | | $1.00 \times 10^{-8}$ | $1.0 \pm 0.1 \times 10^{-8}$ amperes |
| 1.% | ( D ) | | $1.00 \times 10^{-7}$ | $1.0 \pm 0.1 \times 10^{-7}$ amperes |
| 10.% | ( D ) | | $1.00 \times 10^{-6}$ | $1.0 \pm 0.1 \times 10^{-6}$ amperes |
| 100.% | ( D ) | | $1.00 \times 10^{-5}$ | $1.0 \pm 0.1 \times 10^{-5}$ amperes |
| Sample Flow | ( D ) | — | 28.3 SLPM | 28.3 ± 2.8 SLPM |

* Internal Ref refers to a known concentration level and has no effect upon instrument operation.

( A ) In tolerance when received    ( B ) Out of tolerance when received    ( C ) Inoperable

( D ) As found results not applicable - new instrument ☐ TOLERANCE NOT APPLICABLE TO THIS INSTRUMENT.

## MAINTENANCE PERFORMED

| | | | |
|---|---|---|---|
| ☐ Rework Scattering Chamber | ☐ Replace Standard Lamp | ☐ Test Absolute Filter | ☐ Align Optics |
| ☐ Replace Smoke Chamber | ☐ Test Electrical Connections | ☐ Replace Gaskets | ☐ |
| ☐ Clean Sampling System | ☐ Perform Voltage Measurements | ☐ Tighten Loose Hardware | ☐ |
| ☐ Replace Cell Lamp | ☐ Flush Vacuum Pump | ☐ Leak Check | ☒ Final Test |
| ☐ Adjust Internal Reference | ☐ Test Scanning Probe | ☐ Align Amplifier | ☒ Final Inspection KK |

## CALIBRATION PROCEDURES USED

TEMP: 22.7 °C    R.H.: 36 %

| | | | |
|---|---|---|---|
| ☐ ATI-0001 | ☐ ATI-0004 | ☐ ATI-0006 | ☐ ATI-0036A |
| ☐ ATI-0003 | ☐ ATI-0005 | ☒ ATI-0007 | ☐ |

CALIBRATED BY: [signature]    B30    DATE: 7 Dec 92

QA REVIEW:    REVISED: 4/21 DWC

# Met One

**Met One, Inc.**
481 California Avenue
Grants Pass, OR 97526

Telephone: (503) 479-1248
FAX: (503) 479-3057

TWX: 510-755-0774
Cable: MET ONE

REPORT NO. 931869

DATE 6-4-93

## REPORT OF CALIBRATION

MODEL 085A SERIAL NUMBER 93510

This certifies the above named instrument conforms to the original specifications in effect at time of manufacture.

Calibration has been accomplished by comparison with standards maintained by MET ONE. The accuracy and stability of standards maintained by MET ONE are traceable to the National Institute of Standards and Technology, or have been derived from acceptable values of natural physical constants, or have been derived by the ratio type of self calibration.

Calibration was performed at a reference temperature of 69.1°F and a relative humidity of 29.2%.

A record of all work performed is maintained by MET ONE, INC.

Next calibration on this instrument is due 6-4-94.

Signed: David A. Wilson

ASTM 63F Thermometer 21 December, 1992

Fluke 8062A DVM 17 July, 1992

FORM CAL085 Revi (07/24/92) TEP

Shortridge Instruments, Inc.

7855 EAST REDFIELD ROAD / SCOTTSDALE, ARIZONA 85260
TELEPHONE (602) 991-6744 / FAX (602) 443-1267

# CERTIFICATE OF CALIBRATION

INSTRUMENT AirData Multimeter

MODEL ADM-860 SERIAL NO M93068

TEST BY L. Laubmeier DATE 1/22/93

This is to certify that this instrument has been calibrated using instrumentation which is traceable to masters at the National Institute of Standards and Technology, NIST Calibration Certification reference numbers are: differential and absolute pressure: TN-249770-92 dated 2-92; temperature: 88024 dated 10-90.

Quality Assurance Program and calibration procedures meet the requirements for 10CFR 50, Appendix B; 10CFR 21; ANSI/N45.2; and Mil-STD45662A.

CERTIFIED BY: Ernest R. Shortridge

SI

# APPENDIX III

# Test Data and Analysis

A. Test Data

- Testing Series I
- Testing Series II
- Testing Series III

B. "Statistical Analysis of the HEPA Filter Challenge Aerosol Comparison Testing Performed by Eli Lilly and Company and the Joseph Kennedy Company", John R. Murphy, Ph.D., 25 August 1993.

# TESTING SERIES I*

| TEST | FILTER | CHALLENGE AEROSOL | UPSTREAM CONC. (micrograms/ltr) | AIR TEMP (°F) | AIR RH (%) | AIR VEL (fpm) | GEN. | PHOTO. | PENETRATION SCAN READINGS (%) #1 | #2 | #3 | #4 | #5 | #6 | #7 | #8 | #9 | #10 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | #1 | DOP | 11.0 | 83.4 | 41.0 | 355 | A | A | .006 | .09 | .04 | .10 | .003 | .12 | .20 | .092 | .20 | .063 |
| 2 | #1 | DOP | 11.0 | 84.0 | 39.3 | 355 | A | B | .014 | .054 | .032 | .12 | .008 | .12 | .20 | .085 | .20 | .08 |
| 3 | #1 | DOP | 12.0 | 84.1 | 38.6 | 341 | B | B | .008 | .045 | .03 | .14 | .006 | .10 | .20 | .070 | .17 | .078 |
| 4 | #1 | DOP | 12.0 | 84.1 | 38.6 | 371 | B | A | .005 | .042 | .038 | .15 | .006 | .085 | .21 | .058 | .18 | .070 |
| 5 | #1 | Emery 3004 | 12.0 | 85.3 | 37.8 | 356 | A | A | .012 | .035 | .040 | .13 | .005 | .12 | .22 | .072 | .20 | .072 |
| 6 | #1 | Emery 3004 | 12.0 | 85.3 | 36.9 | 374 | A | B | .010 | .055 | .038 | .12 | .006 | .12 | .20 | .085 | .20 | .074 |
| 7 | #1 | Emery 3004 | 12.0 | 85.7 | 36.4 | 348 | B | B | .010 | .048 | .038 | .15 | .008 | .13 | .21 | .075 | .20 | .08 |
| 8 | #1 | Emery 3004 | 12.0 | 85.7 | 36.5 | 362 | B | A | .005 | .044 | .038 | .16 | .005 | .13 | .21 | .082 | .20 | .075 |

Note: Comparative data pairs are Test 1 vs. Test 5 (i.e., Test 1 #10 versus Test 5 #10), Test 2 vs. Test 6, Test 3 vs. Test 7, and Test 4 vs. Test 8.

Photometers and aerosol generators alternated between use of DOP and Emery 3004. Air velocity held within 10% of target 342 fpm. Photometer probe moved between comparative readings. One filter tested.

*Using Test Procedure I

## TESTING SERIES II*

| TEST | FILTER | CHALLENGE AEROSOL | UPSTREAM CONC. (micrograms/ltr) | AIR TEMP (°F) | AIR RH (%) | AIR VEL (fpm) | GEN. | PHOTO. | PENETRATION SCAN READINGS (%) #1 | #2 | #3 | #4 | #5 | #6 | #7 | #8 | #9 | #10 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 9A | #1 | Emery 3004 | 12.0 | 78.4 | 39.4 | 382 | B | A | .009 | .045 | .040 | .15 | .006 | .14 | .23 | .074 | .22 | .072 |
| 9B | #1 | DOP | 11.5 | 78.4 | 39.4 | 382 | A | A | .007 | .044 | .038 | .14 | .004 | .105 | .23 | .064 | .21 | .07 |
| 10A | #2 | Emery 3004 | 12.0 | 79.4 | 40.0 | 386 | B | A | .007 | .038 | .048 | .18 | .018 | .40 | .35 | .048 | .24 | .27 |
| 10B | #2 | DOP | 11.5 | 79.4 | 40.0 | 386 | A | A | .005 | .035 | .044 | .16 | .016 | .38 | .33 | .042 | .24 | .25 |
| 11A | #2 | Emery 3004 | 20.0 | 81.7 | 39.1 | 514 | B | A | .005 | .029 | .035 | .15 | .015 | .28 | .34 | .028 | .082 | .18 |
| 11B | #2 | DOP | 18.0 | 81.7 | 39.1 | 514 | A | A | .004 | .028 | .033 | .15 | .013 | .26 | .33 | .025 | .074 | .16 |
| 12A | #2 | DOP | 17.0 | 82.6 | 38.6 | 260 | A | A | .005 | .055 | .035 | .10 | .008 | .30 | .29 | .020 | .058 | .21 |
| 12B | #2 | Emery 3004 | 18.0 | 82.6 | 38.6 | 260 | B | A | .006 | .061 | .040 | .11 | .009 | .30 | .32 | .023 | .062 | .22 |
| 17A | #3 | Emery 3004 | 18.0 | 78.5 | 39.0 | 59 | B | B | .010 | .022 | .004 | .27 | .018 | .022 | .36 | .007 | .22 | .076 |
| 17B | #3 | DOP | 14.0 | 78.5 | 39.0 | 59 | A | B | .014 | .023 | .005 | .23 | .016 | .02 | .35 | .003 | .22 | .074 |
| 18A | #3 | DOP | 17.0 | 84.5 | 41.3 | 106 | A | B | .009 | .015 | .007 | .40 | .016 | .005 | .13 | .068 | .27 | .004 |
| 18B | #3 | Emery 3004 | 19.0 | 84.5 | 41.3 | 106 | B | B | .008 | .014 | .006 | .40 | .017 | .007 | .13 | .07 | .26 | .005 |

Note: Comparative data points are the "A" series and "B" series for a trial run (i.e., 9A #4 versus 9B #4).

Single photometer used for comparative tests. Generators dedicated to challenge aerosol type. Air velocity varied. Three filters tested. Photometer probe fixed between comparative readings.

*Using Test Procedure II

## TESTING SERIES III*

| TEST | FILTER | CHALLENGE AEROSOL | UPSTREAM CONC. (micrograms/ltr) | AIR TEMP (°F) | AIR RH (%) | AIR VEL (fpm) | GEN. | PHOTO. | PENETRATION SCAN READINGS (%) #1 | PENETRATION SCAN READINGS (%) #9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 13A | #1 | Emery/DOP/Emery/DOP | 18.0 (Emery) | 84.9 | 45.8 | 233 | B | A | .008/.007/.008/.008 | .15/.14/.15/.14 |
| 13B | #1 | | 17.5 (DOP) | 84.9 | 45.8 | 233 | A | A | | |
| 14A | #1 | DOP/Emery/DOP/Emery | 18.0 (DOP) | 86.0 | 42.7 | 231 | A | A | .007/.008/.007/.008 | .14/.16/.15/.16 |
| 14B | #1 | | 19.5 (Emery) | 86.0 | 42.7 | 231 | B | A | | |
| 15A | #1 | Emery/DOP/Emery/DOP | 13.0 (Emery) | 83.2 | 40.3 | 359 | B | A | .007/.005/.005/.005 | .18/.16/.17/.16 |
| 15B | #1 | | 12.0 (DOP) | 83.2 | 40.3 | 359 | A | A | | |
| 16A | #1 | DOP/Emery/DOP/Emery | 12.0 (DOP) | 83.2 | 40.3 | 359 | A | A | .005/.006/.006/.005 | .16/.16/.16/.16 |
| 16B | #1 | | 13.0 (Emery) | 83.2 | 40.3 | 359 | B | A | | |

Note: Comparative data points are sequential in the testing (i.e., for Tests 13A and 13B; comparative data are .008 versus .007, .008 versus .008, .15 versus .14 and so forth).

Single photometer used for comparative tests. Generators dedicated to challenge aerosol type. Air velocity varied. One filter tested. Photometer probe fixed between comparative readings.

*Using Test Procedure III

## Statistical Analysis of the HEPA Filter Challenge Aerosol Comparison Testing Performed by Eli Lilly and Company and the Joseph Kennedy Company

Data from three series of runs have been statistically analyzed. Conclusions are based on geometric statistics resulting from the analysis of the logarithms of the measurements.

The first series of eight runs were performed by shutting down the equipment before moving the probe from one pinhole location to the next on the filter. Also in this series, Runs 1-4 were performed using DOP aerosol, while Runs 5-8 used Emery aerosol. The second series of Runs 9-12 & 17-18, on the other hand, were performed by switching aerosol at each pinhole in each run before moving on to the next location on the filter. Runs 17-18 were performed later and separately from Runs 9-12 using a different filter and setup to achieve lower flow rates. The second series provided a direct comparison of the aerosols at each pinhole in each run. The third series of Runs 13-16 was to test whether switching back and forth between the aerosol types at each pinhole had any noticeable effect on the level detected due to possible buildup and/or carryover. In this series, Runs 13 & 15 used crossover sequence 1 : Emery - DOP - Emery - DOP, while Runs 14 & 16 used crossover sequence 2 : DOP - Emery - DOP - Emery.

The analyses found that the effect of different equipment and/or conditions in the various runs was mostly insignificant. Equipment changes included different smoke generators and different probes, while testing conditions included different air velocities and slightly different upstream concentrations. The analyses did find a difference in downstream concentration between Runs 9-12 and Runs 17-18 in the second series. This difference could be due to different filters, different setup configurations, or different air velocities. Also, in the second series, there was some indication that the ratio of Emery to DOP percent aerosol penetration was related to the size of pinhole, but this phenomenon was not apparent in any of the other series, and such an effect is therefore deemed unlikely to have any significance under normal testing conditions.

Overall, there is a good correspondence between the measurement results of the two aerosol types. When the results for one are plotted against results for the other on a logarithmic scale, the correlation coefficient is 0.995.

In all but the first series, the average percent penetration using Emery was statistically higher than the corresponding average using DOP. The average ratios were:

| | |
|---|---|
| Series I Runs 1-8: | Ratio of Emery to DOP = 1.057 (n=10) |
| Series II Runs 9-12: | Ratio of Emery to DOP = 1.117 (n=40) |
| Series II Runs 17-18: | Ratio of Emery to DOP = 1.082 (n=20) |
| Series III Runs 13-16: | Ratio of Emery to DOP = 1.090 (n=8) |

The overall weighted average ratio of Emery to DOP is 1.083, with a standard error of .022. Hence, we may say with 95% confidence that the average ratio of Emery to DOP is somewhere between 1.040 and 1.126.

In summary, the three series of tests provide evidence that Emery and DOP aerosols are comparable in the downstream concentrations they obtain over a wide range of operating conditions. The tests also give a good indication that Emery aerosol is slightly more sensitive than DOP aerosol for measuring downstream concentrations. For the same upstream concentration, downstream concentrations using Emery aerosol can be expected to average about 8% ± 4% higher than those using DOP aerosol.

John R. Murphy, Statistical and Mathematical Sciences, Eli Lilly and Company
08/25/93

Attachments

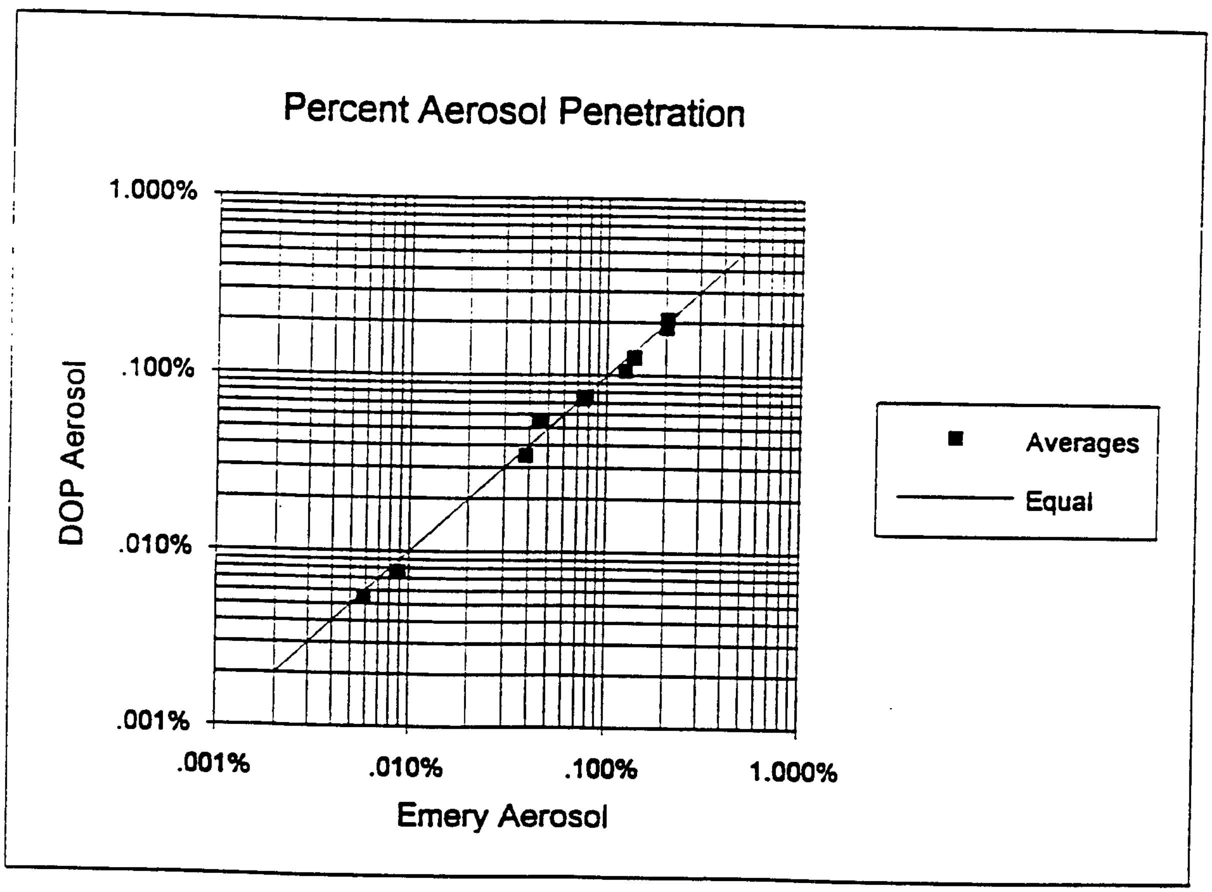
HEPA Filter Challenge Aerosol Comparison Testing
Eli Lilly and Company and Joseph Kennedy Company

First Series (Runs 1-8)

| Run Number | Aerosol Type | Pinhole Size (gage) 16 | 18 | 20 | 21 | 22 | 23 | 25 | 26 | 27 | 30 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | DOP | .200% | .210% | .063% | .100% | .090% | .120% | .092% | .040% | .003% | .006% |
| 2 | DOP | .200% | .210% | .080% | .120% | .054% | .120% | .085% | .032% | .008% | .014% |
| 3 | DOP | .170% | .200% | .078% | .140% | .045% | .100% | .070% | .030% | .006% | .008% |
| 4 | DOP | .180% | .220% | .070% | .150% | .042% | .085% | .058% | .038% | .006% | .005% |
| 5 | Emery | .200% | .210% | .072% | .130% | .035% | .120% | .072% | .040% | .005% | .012% |
| 6 | Emery | .200% | .200% | .074% | .120% | .055% | .120% | .085% | .038% | .006% | .010% |
| 7 | Emery | .200% | .200% | .080% | .150% | .048% | .130% | .075% | .038% | .008% | .010% |
| 8 | Emery | .200% | .200% | .075% | .160% | .044% | .130% | .082% | .038% | .005% | .005% |

6/22/93 — Filter 1 (T-007-8-05-05-IU)

| | | | |
|---|---|---|---|
| Run 1: | Avg. Velocity 355 fpm – Gen. A, Sensor A | Run 5: | Avg. Velocity 356 fpm – Gen. A, Sensor A |
| Run 2: | Avg. Velocity 355 fpm – Gen. A, Sensor B | Run 6: | Avg. Velocity 374 fpm – Gen. A, Sensor B |
| Run 3: | Avg. Velocity 341 fpm – Gen. B, Sensor B | Run 7: | Avg. Velocity 348 fpm – Gen. B, Sensor B |
| Run 4: | Avg. Velocity 371 fpm – Gen. B, Sensor A | Run 8: | Avg. Velocity 362 fpm – Gen. B, Sensor A |

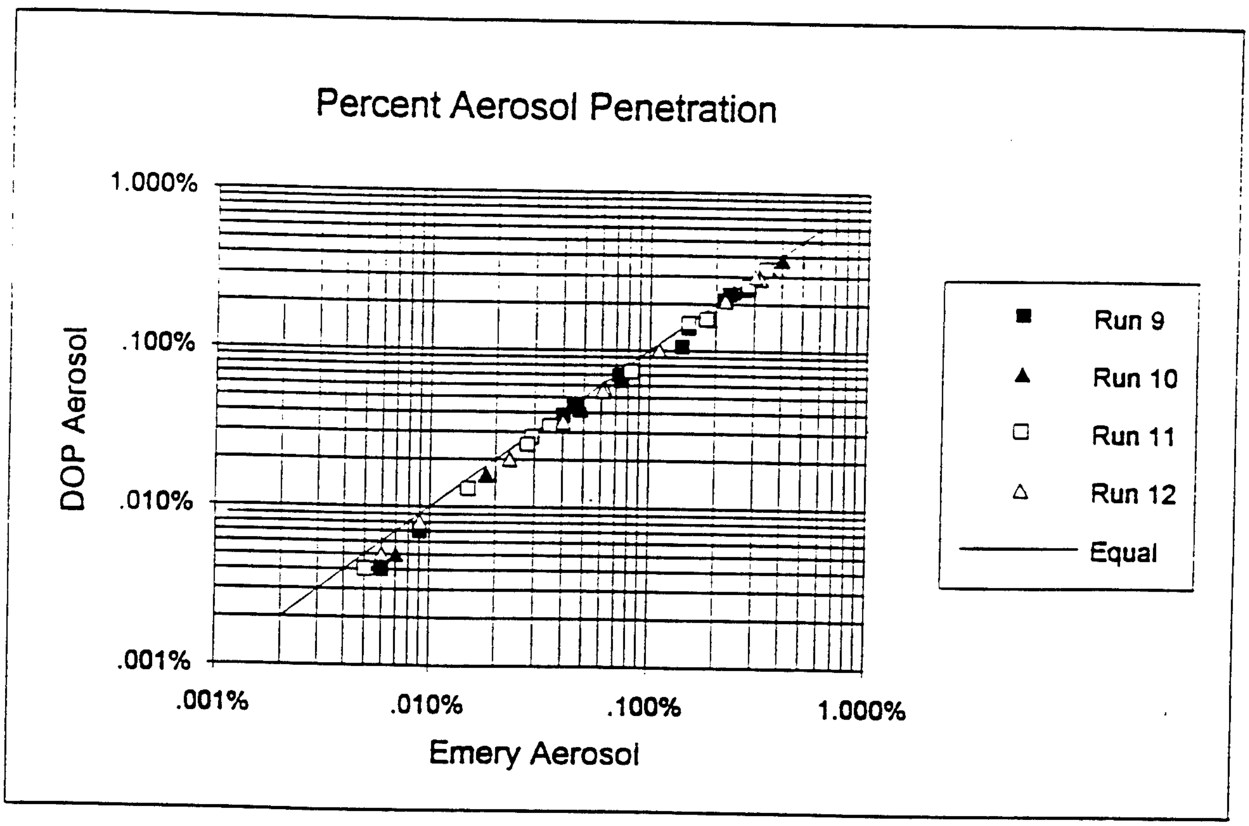


## HEPA Filter Challenge Aerosol Comparison Testing
## Eli Lilly and Company and Joseph Kennedy Company

### Second Series (Runs 9-12)

| Run Number | Aerosol Type | Pinhole Size (gage) 16 | 18 | 20 | 21 | 22 | 23 | 25 | 26 | 27 | 30 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 9A | Emery | .220% | .230% | .072% | .150% | .045% | .140% | .074% | .040% | .006% | .009% |
| 9B | DOP | .210% | .230% | .070% | .140% | .044% | .105% | .064% | .038% | .004% | .007% |
| 9 | Ratio | 1.048 | 1.000 | 1.029 | 1.071 | 1.023 | 1.333 | 1.156 | 1.053 | 1.500 | 1.286 |
| 10A | Emery | .240% | .350% | .270% | .180% | .038% | .400% | .048% | .048% | .018% | .007% |
| 10B | DOP | .240% | .330% | .250% | .160% | .035% | .380% | .042% | .044% | .016% | .005% |
| 10 | Ratio | 1.000 | 1.061 | 1.080 | 1.125 | 1.086 | 1.053 | 1.143 | 1.091 | 1.125 | 1.400 |
| 11A | Emery | .082% | .340% | .180% | .150% | .029% | .280% | .028% | .035% | .015% | .005% |
| 11B | DOP | .074% | .330% | .160% | .150% | .028% | .260% | .025% | .033% | .013% | .004% |
| 11 | Ratio | 1.108 | 1.030 | 1.125 | 1.000 | 1.036 | 1.077 | 1.120 | 1.061 | 1.154 | 1.250 |
| 12B | Emery | .062% | .320% | .220% | .110% | .061% | .300% | .023% | .040% | .009% | .006% |
| 12A | DOP | .058% | .290% | .210% | .100% | .055% | .300% | .020% | .035% | .008% | .005% |
| 12 | Ratio | 1.069 | 1.103 | 1.048 | 1.100 | 1.109 | 1.000 | 1.150 | 1.143 | 1.125 | 1.200 |

| | |
|---|---|
| Run 9: | 6/24/93 — Filter 1 (T-007-8-05-05-IU) — Average Velocity 382 fpm |
| Run 10: | 6/24/93 — Filter 2 (AAF 1431915-539) — Average Velocity 386 fpm |
| Run 11: | 6/24/93 — Filter 2 (AAF 1431915-539) — Average Velocity 514 fpm |
| Run 12: | 6/24/93 — Filter 2 (AAF 1431915-539) — Average Velocity 260 fpm |





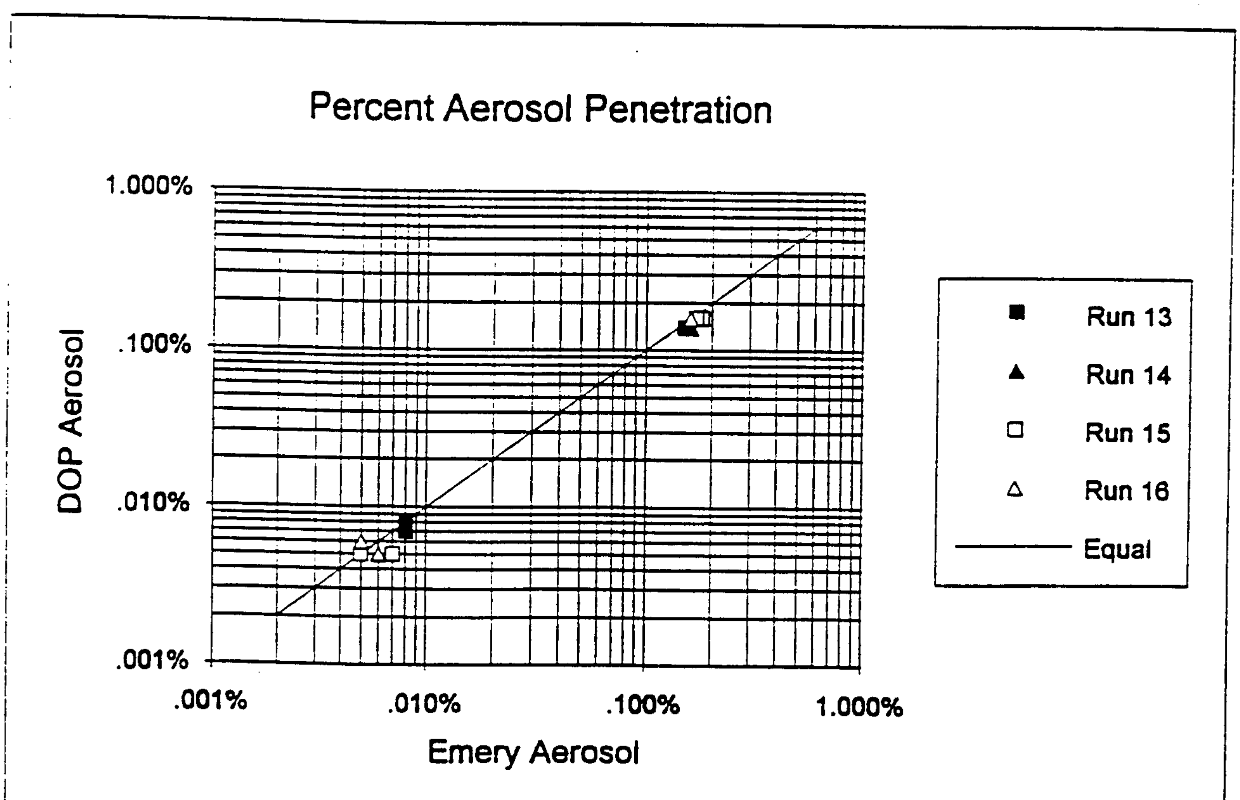

8/25/93

## HEPA Filter Challenge Aerosol Comparison Testing
## Eli Lilly and Company and Joseph Kennedy Company

### Third Series (Runs 13-16)

| Run Number | Aerosol Type | Pinhole Size (gage) | | | | | |
|---|---|---|---|---|---|---|---|
| | | 16(1) | 16(2) | 16(avg) | 30(1) | 30(2) | 30(avg) |
| 13A | Emery | .150% | .150% | .1500% | .008% | .008% | .0080% |
| 13B | DOP | .140% | .140% | .1400% | .007% | .008% | .0075% |
| 13 | Ratio | 1.071 | 1.071 | 1.071 | 1.143 | 1.000 | 1.067 |
| 14B | Emery | .160% | .160% | .1600% | .008% | .008% | .0080% |
| 14A | DOP | .140% | .140% | .1400% | .007% | .007% | .0070% |
| 14 | Ratio | 1.143 | 1.143 | 1.143 | 1.143 | 1.143 | 1.143 |
| 15A | Emery | .180% | .170% | .1750% | .007% | .005% | .0060% |
| 15B | DOP | .160% | .160% | .1600% | .005% | .005% | .0050% |
| 15 | Ratio | 1.125 | 1.063 | 1.094 | 1.400 | 1.000 | 1.200 |
| 16B | Emery | .160% | .160% | .1600% | .006% | .005% | .0055% |
| 16A | DOP | .160% | .160% | .1600% | .005% | .006% | .0055% |
| 16 | Ratio | 1.000 | 1.000 | 1.000 | 1.200 | 0.833 | 1.000 |

| | |
|---|---|
| Run 13: | 7/9/93 — Filter 1 (T-007-8-05-05-IU) — Sequence 1: (E - D - E - D) |
| Run 14: | 7/9/93 — Filter 1 (T-007-8-05-05-IU) — Sequence 2: (D - E - D - E) |
| Run 15: | 7/14/93 — Filter 1 (T-007-8-05-05-IU) — Sequence 1: (E - D - E - D) |
| Run 16: | 7/14/93 — Filter 1 (T-007-8-05-05-IU) — Sequence 2: (D - E - D - E) |





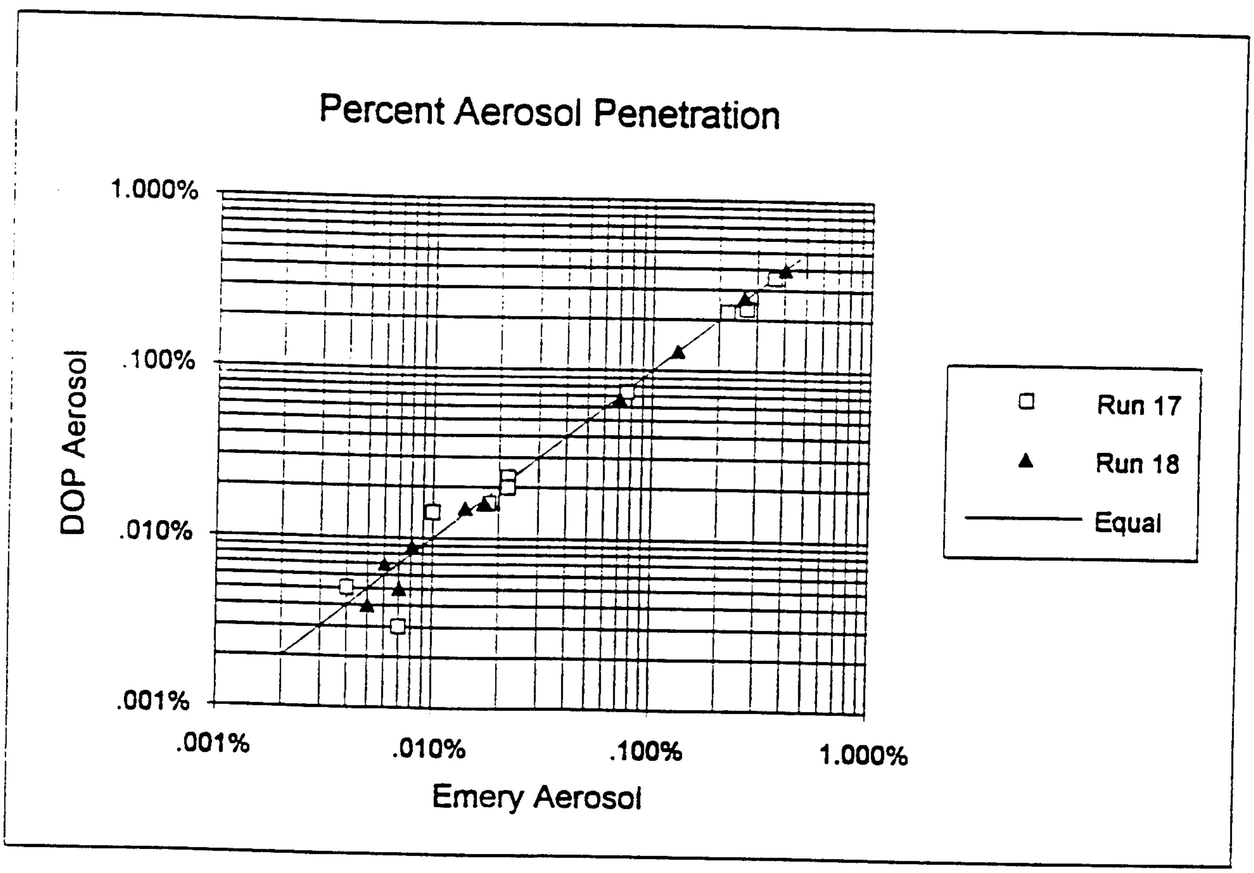

8/25/93

HEPA Filter Challenge Aerosol Comparison Testing
Eli Lilly and Company and Joseph Kennedy Company

Second Series (Runs 17-18)

| Run Number | Aerosol Type | Pinhole Size (gage) 16 | 18 | 20 | 21 | 22 | 23 | 25 | 26 | 27 | 30 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 17A | Emery | .220% | .360% | .076% | .270% | .022% | .022% | .007% | .004% | .018% | .010% |
| 17B | DOP | .220% | .350% | .074% | .230% | .023% | .020% | .003% | .005% | .016% | .014% |
| 17 | Ratio | 1.000 | 1.029 | 1.027 | 1.174 | 0.957 | 1.100 | 2.333 | 0.800 | 1.125 | 0.714 |
| 18B | Emery | .260% | .130% | .005% | .400% | .014% | .007% | .070% | .006% | .017% | .008% |
| 18A | DOP | .270% | .130% | .004% | .400% | .015% | .005% | .068% | .007% | .016% | .009% |
| 18 | Ratio | 0.963 | 1.000 | 1.250 | 1.000 | 0.933 | 1.400 | 1.029 | 0.857 | 1.063 | 0.889 |

| | |
|---|---|
| Run 17: | 7/21/93 — Filter 3 (0-007-2-19-06-SU) — Average Velocity 59 fpm |
| Run 18: | 7/26/93 — Filter 3 (0-007-2-19-06-SU) — Average Velocity 106 fpm |

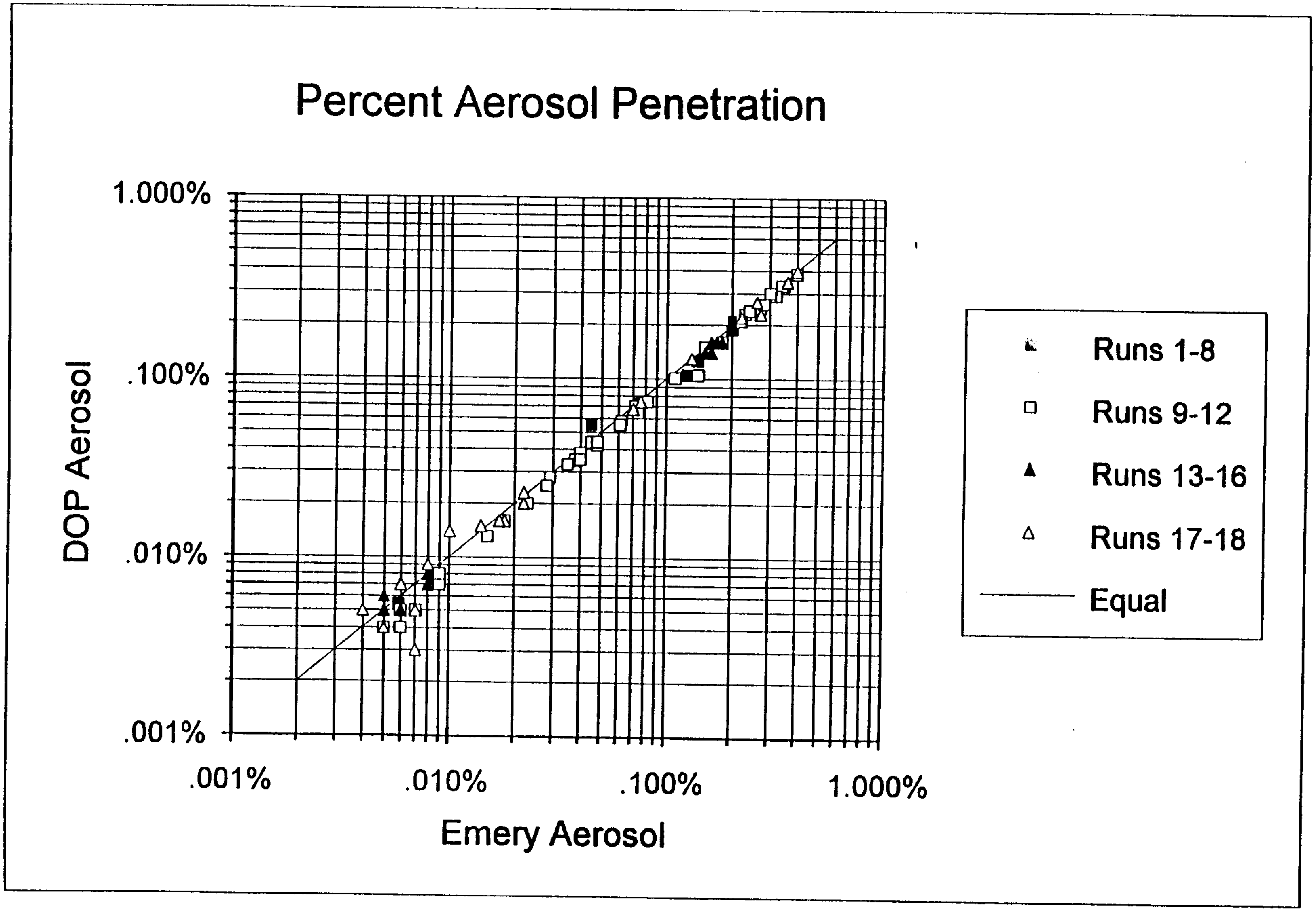





'25/93

HEPA Filter Challenge Aerosol Comparison Testing
Eli Lilly and Company and Joseph Kennedy Company

All Test Runs Combined




9/8/93

First Series - Runs 1-8

General Linear Models Procedure
Class Level Information

| Class | Levels | Values |
|---|---|---|
| TYPE | 2 | DOP Emery |
| RUN | 8 | 1 2 3 4 5 6 7 8 |
| GAGE | 10 | 16 18 20 21 22 23 25 26 27 30 |

Number of observations in data set = 80

Dependent Variable: LOGPCT

| Source | DF | Sum of Squares | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| Model | 25 | 111.116652 | 4.444666 | 100.80 | 0.0001 |
| Error | 54 | 2.381018 | 0.044093 | | |
| Corrected Total | 79 | 113.497670 | | | |

| R-Square | C.V. | Root MSE | LOGPCT Mean |
|---|---|---|---|
| 0.979021 | -7.271820 | 0.20998 | -2.88763 |

| Source | DF | Type I SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| TYPE | 1 | 0.051433 | 0.051433 | 1.17 | 0.2849 |
| RUN(TYPE) | 6 | 0.318375 | 0.053063 | 1.20 | 0.3188 |
| GAGE | 9 | 110.542269 | 12.282474 | 278.56 | 0.0001 |
| TYPE*GAGE | 9 | 0.204574 | 0.022730 | 0.52 | 0.8569 |

| Source | DF | Type III SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| TYPE | 1 | 0.051433 | 0.051433 | 1.17 | 0.2849 |
| RUN(TYPE) | 6 | 0.318375 | 0.053063 | 1.20 | 0.3188 |
| GAGE | 9 | 110.542269 | 12.282474 | 278.56 | 0.0001 |
| TYPE*GAGE | 9 | 0.204574 | 0.022730 | 0.52 | 0.8569 |

Second Series - Runs 9-12 & 17-18

General Linear Models Procedure
Class Level Information

| Class | Levels | Values |
|---|---|---|
| FILTER | 3 | 1 2 3 |
| RUN | 6 | 9 10 11 12 17 18 |
| TYPE | 2 | DOP Emery |
| GAGE | 10 | 16 18 20 21 22 23 25 26 27 30 |

Number of observations in data set = 120

Dependent Variable: LOGPCT

| Source | DF | Sum of Squares | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| Model | 74 | 250.600945 | 3.386499 | 276.83 | 0.0001 |
| Error | 45 | 0.550483 | 0.012233 | | |
| Corrected Total | 119 | 251.151428 | | | |

| R-Square | C.V. | Root MSE | LOGPCT Mean |
|---|---|---|---|
| 0.997808 | -3.663801 | 0.11060 | -3.01880 |

| Source | DF | Type I SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| FILTER | 2 | 11.738478 | 5.869239 | 479.79 | 0.0001 |
| RUN(FILTER) | 3 | 2.233855 | 0.744618 | 60.87 | 0.0001 |
| GAGE | 9 | 163.618582 | 18.179842 | 1486.14 | 0.0001 |
| FILTER*GAGE | 18 | 51.673821 | 2.870768 | 234.68 | 0.0001 |
| RUN*GAGE(FILTER) | 27 | 20.931197 | 0.775230 | 63.37 | 0.0001 |
| TYPE | 1 | 0.224716 | 0.224716 | 18.37 | 0.0001 |
| FILTER*TYPE | 2 | 0.027320 | 0.013660 | 1.12 | 0.3363 |
| RUN*TYPE(FILTER) | 3 | 0.004692 | 0.001564 | 0.13 | 0.9431 |
| TYPE*GAGE | 9 | 0.148284 | 0.016476 | 1.35 | 0.2408 |

| Source | DF | Type III SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| FILTER | 2 | 11.738478 | 5.869239 | 479.79 | 0.0001 |
| RUN(FILTER) | 3 | 2.233855 | 0.744618 | 60.87 | 0.0001 |
| GAGE | 9 | 150.579328 | 16.731036 | 1367.70 | 0.0001 |
| FILTER*GAGE | 18 | 51.673821 | 2.870768 | 234.68 | 0.0001 |
| RUN*GAGE(FILTER) | 27 | 20.931197 | 0.775230 | 63.37 | 0.0001 |
| TYPE | 1 | 0.219934 | 0.219934 | 17.98 | 0.0001 |
| FILTER*TYPE | 2 | 0.027320 | 0.013660 | 1.12 | 0.3363 |
| RUN*TYPE(FILTER) | 3 | 0.004692 | 0.001564 | 0.13 | 0.9431 |
| TYPE*GAGE | 9 | 0.148284 | 0.016476 | 1.35 | 0.2408 |

Third Series - Runs 13-16

General Linear Models Procedure
Class Level Information

| Class | Levels | Values |
|---|---|---|
| SEQUENCE | 2 | 1 2 |
| RUN | 4 | 13 14 15 16 |
| TYPE | 2 | DOP Emery |
| GAGE | 2 | 16 30 |

Number of observations in data set = 16

Dependent Variable: LOGPCT

| Source | DF | Sum of Squares | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| Model | 12 | 40.6965461 | 3.3913788 | 6103.60 | 0.0001 |
| Error | 3 | 0.0016669 | 0.0005556 | | |
| Corrected Total | 15 | 40.6982130 | | | |

| R-Square | C.V. | Root MSE | LOGPCT Mean |
|---|---|---|---|
| 0.999959 | -0.682820 | 0.02357 | -3.45214 |

| Source | DF | Type I SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| SEQUENCE | 1 | 0.0004598 | 0.0004598 | 0.83 | 0.4301 |
| RUN(SEQUENCE) | 2 | 0.0496298 | 0.0248149 | 44.66 | 0.0059 |
| TYPE | 1 | 0.0282684 | 0.0282684 | 50.88 | 0.0057 |
| SEQUENCE*TYPE | 1 | 0.0011972 | 0.0011972 | 2.15 | 0.2384 |
| RUN*TYPE(SEQUENCE) | 2 | 0.0113097 | 0.0056549 | 10.18 | 0.0460 |
| GAGE | 1 | 40.4146643 | 40.4146643 | 72735.83 | 0.0001 |
| SEQUENCE*GAGE | 1 | 0.0000793 | 0.0000793 | 0.14 | 0.7307 |
| RUN*GAGE(SEQUENCE) | 2 | 0.1904508 | 0.0952254 | 171.38 | 0.0008 |
| TYPE*GAGE | 1 | 0.0004868 | 0.0004868 | 0.88 | 0.4183 |

| Source | DF | Type III SS | Mean Square | F Value | Pr > F |
|---|---|---|---|---|---|
| SEQUENCE | 1 | 0.0004598 | 0.0004598 | 0.83 | 0.4301 |
| RUN(SEQUENCE) | 2 | 0.0496298 | 0.0248149 | 44.66 | 0.0059 |
| TYPE | 1 | 0.0282684 | 0.0282684 | 50.88 | 0.0057 |
| SEQUENCE*TYPE | 1 | 0.0011972 | 0.0011972 | 2.15 | 0.2384 |
| RUN*TYPE(SEQUENCE) | 2 | 0.0113097 | 0.0056549 | 10.18 | 0.0460 |
| GAGE | 1 | 40.4146643 | 40.4146643 | 72735.83 | 0.0001 |
| SEQUENCE*GAGE | 1 | 0.0000793 | 0.0000793 | 0.14 | 0.7307 |
| RUN*GAGE(SEQUENCE) | 2 | 0.1904508 | 0.0952254 | 171.38 | 0.0008 |
| TYPE*GAGE | 1 | 0.0004868 | 0.0004868 | 0.88 | 0.4183 |